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FINAL SAMPLING AND ANALYSIS PLAN AND QUALITY ASSURANCE PROJECT PLAN FOR REMEDIAL INVESTIGATION POTENTIAL SOURCE OF CONTAMINATION 45 (PSC45) WITH TRANSMITTAL NAS JACKSONVILLE FL

6/3/2011

TETRA TECH

TETRA TECH

Document Tracking Number 11JAX0133

June 3, 2011

Project Number 112G02686

Ms. Adrienne Wilson
Remedial Project Manager
Code OPDE3/AW
Department of the Navy
Naval Facilities Southeast
ATTN: Ajax Street, Building 135N
P.O. Box 30A
NAS Jacksonville, FL 32212-0030

Reference: CLEAN V Contract Number N62467-08-D-1001
Contract Task Order Number JM19

Subject: Final Sampling and Analysis Plan (SAP) (Field Sampling Plan and Quality Assurance Project Plan) for Remedial Investigation for Potential Source of Contamination 45, Naval Air Station Jacksonville Jacksonville, Florida

Dear Ms. Wilson:

Tetra Tech NUS, Inc. (Tetra Tech) is pleased to present the Final Sampling and Analysis Plan (SAP) (Field Sampling Plan and Quality Assurance Project Plan) for Remedial Investigation for Potential Source of Contamination (PSC) 45, Naval Air Station Jacksonville, Florida for your approval. Comments to this document have been received from the NAS Jacksonville Partnering team and the Response to Comments is enclosed. A copy of this document has also been forwarded to the NAS Jacksonville Partnering Team members listed below.

If you have any questions regarding the enclosed material, or if I can be of assistance in any way, please contact me at (904) 730-4669, extension 213, or by e-mail at Mark.Peterson@tetratech.com.

Sincerely,

Mark A. Peterson
Task Order Manager

Enclosures (2)

c: Tim Curtin, NAS Jacksonville (hard copy/CD)
Pete Dao, USEPA (hard copy/CD)
David Grabka, FDEP (hard copy/CD)
John Trepanowski, Tetra Tech (cover letter only)
CTO JM19 Project File (2 copies)

Tetra Tech NUS, Inc.
8640 Philips Highway, Suite 16, Jacksonville, Florida 32256
Tel 904.636.6125 Fax 904.636.6165 www.tetratech.com

Comprehensive Long-term Environmental Action Navy

CONTRACT NUMBER N62470-08-D-1001

Rev. 1
May 2011

Sampling and Analysis Plan
(Field Sampling Plan and Quality Assurance Project Plan)

Remedial Investigation
for
Potential Source of Contamination 45
Former Building 200 Wash Rack Disposal Pit

Naval Air Station Jacksonville
Jacksonville, Florida

Contract Task Order JM19

May 2011

NAVFAC Naval Facilities Engineering Command
NAVFAC SOUTHEAST

NAS Jacksonville
Jacksonville, Florida 32212-0030

TETRA TECH NUS, Inc.

SAP Worksheet #1 – Title and Approval Page
(UFP-QAPP Manual Section 2.1)

SAMPLING AND ANALYSIS PLAN
(Field Sampling Plan and Quality Assurance Project Plan)
May 2011

Remedial Investigation
for
Potential Source of Contamination (PSC) 45
Former Building 200 Wash Rack Disposal Pit
Naval Air Station Jacksonville
Jacksonville, Florida

Prepared for:
Naval Facilities Engineering Command Southeast
Naval Air Station Jacksonville, Building 903
Jacksonville, Florida 32212-0030

Prepared by:
Tetra Tech NUS, Inc.
234 Mall Boulevard
King of Prussia, Pennsylvania 19406-2954

Prepared under:
Comprehensive Long-term Environmental Action Navy (CLEAN) Contract No. N62470-08-D-1001
Contract Task Order JM19

SAP Worksheet #1 - Approval Page
(UFP-QAPP Manual Section 2.1)

Document Title: Sampling and Analysis Plan, (Field Sampling Plan and Quality Assurance Project Plan), Remedial Investigation for Potential Source of Contamination (PSC) 45 – Former Building 200 Wash Rack Disposal Pit, Naval Air Station (NAS) Jacksonville, Florida

Lead Organization: Naval Facilities Engineering Command (NAVFAC) Southeast

Preparer's Name and Organizational Affiliation: Brad Peebles, Tetra Tech NUS, Inc.

Preparer's Address and Telephone Number: 5601 Mariner Street , Suite 490
Tampa, Florida 33609
(813) 282-7890 Ext. 1015

Preparation Date (Day/Month/Year): October 2010

Review Signatures:

Investigative Organization's Project Manager:

Signature/Date April 5, 2011
Mark A. Peterson, Tetra Tech NUS, Inc.

Investigative Organization's Project QA Manager: T.E. Johnston 4-5-2011

Signature/Date
Tom Johnston, Ph.D.,
Tetra Tech NUS, Inc.

Lead Organization's Project Manager:

Signature/Date
Adrienne Wilson, NAVFAC Southeast
Remedial Project Manager

Lead Organization QA Officer/Chemist: Jonathan Tucker Digitally signed by TUCKER.JONATHAN.P.1239524180 Date: 2011.04.25 23:18:20 -04'00'

Signature/Date
Quality Assurance Officer/Chemist, NAVFAC Atlantic

Approval Signatures:

Signature/Date
Peter Dao, USEPA Region 4
Remedial Project Manager

Signature/Date
David Grabka, FDEP
Remedial Project Manager

SAP Worksheet #1 - Approval Page
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Document Title: Sampling and Analysis Plan, (Field Sampling Plan and Quality Assurance Project Plan), Remedial Investigation for Potential Source of Contamination (PSC) 45 – Former Building 200 Wash Rack Disposal Pit, Naval Air Station (NAS) Jacksonville, Florida

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Investigative Organization's Project Manager:

Signature/Date
Mark A. Peterson, Tetra Tech NUS, Inc.

Investigative Organization's Project QA Manager:

Signature/Date
Tom Johnston, Ph.D.,
Tetra Tech NUS, Inc.

Lead Organization's Project Manager: 6/9/11

Signature/Date
Adrienne Wilson, NAVFAC Southeast
Remedial Project Manager

Lead Organization QA Officer/Chemist:

Signature/Date
Quality Assurance Officer/Chemist, NAVFAC Atlantic

Approval Signatures:

6/3/11

Signature/Date
Peter Dao, USEPA Region 4
Remedial Project Manager

6/3/11

Signature/Date
David Grabka, FDEP
Remedial Project Manager

EXECUTIVE SUMMARY

Tetra Tech NUS, Inc. (Tetra Tech) has prepared this Sampling and Analysis Plan (SAP) that encompasses Field Sampling Plan and Quality Assurance Project Plan requirements for the former Building 200 Wash Rack Disposal Pit, Potential Source of Contamination (PSC) 45, Naval Air Station (NAS) Jacksonville located in Jacksonville, Florida under the Comprehensive Long-term Environmental Action Navy (CLEAN) Contract No. N62470-08-D-1001, Contract Task Order (CTO) JM19. Figure ES-1 presents the PSC 45 Site Location Map depicting the location of PSC 45 at NAS Jacksonville.

The former Building 200 Wash Rack Disposal Pit was discovered in 1991 during repair work to correct plumbing problems at Building 200. This pit, which was identified as PSC 45 by NAS Jacksonville in 1991, was a cylindrical concrete lined pit with a soil bottom, located east of the Building 200 Wash Rack. In the past, air station ground support equipment was cleaned and stripped of paint in the wash rack. Effluent from the wash rack discharged into an oil-water separator located below the wash rack. For an unknown period of time the oil-water separator discharged overflow via an underground pipeline to the Building 200 Wash Rack Disposal Pit. After this pit was discovered, the connection from the oil-water separator to the pit was plugged. In July 1991, water, oil, and paint chips were observed in the pit. Shortly thereafter, waste from the pit was removed and disposed of as hazardous waste.

In 1998, the liquid and solids within the pit and the soil surrounding and underlying the pit were removed. Pre-disposal samples collected from the soil, liquid, and solids were analyzed for Toxicity Characteristic Leaching Procedure (TCLP) analytes for waste characterization and the reported concentrations were less than the applicable TCLP regulatory limits. No post-excavation sampling around the former pit has been documented.

A new oil/water separator was installed within the excavation area. The old oil/water separator is still operational in the wash rack room; however, to further safeguard against oil and solvents being released to the environment, effluent from the old separator is directed through plumbing to the new separator which then flows to the sanitary sewer. Currently, the area from where the disposal pit was removed is covered by a 4-foot by 12-foot concrete pad.

Soil and groundwater samples were collected in August 2009 during the Site Assessment (SA) for PSC 45. Soil samples, which were collected next to the new oil/water separator, were analyzed for volatile organic compounds (VOCs), semivolatile organic compounds (SVOCs), polychlorinated biphenyls (PCBs), total recoverable petroleum hydrocarbons (TRPH), and metals. Groundwater samples were collected along the west, north, and east sides of Building 200 and analyzed by a mobile laboratory for VOCs and naphthalene.

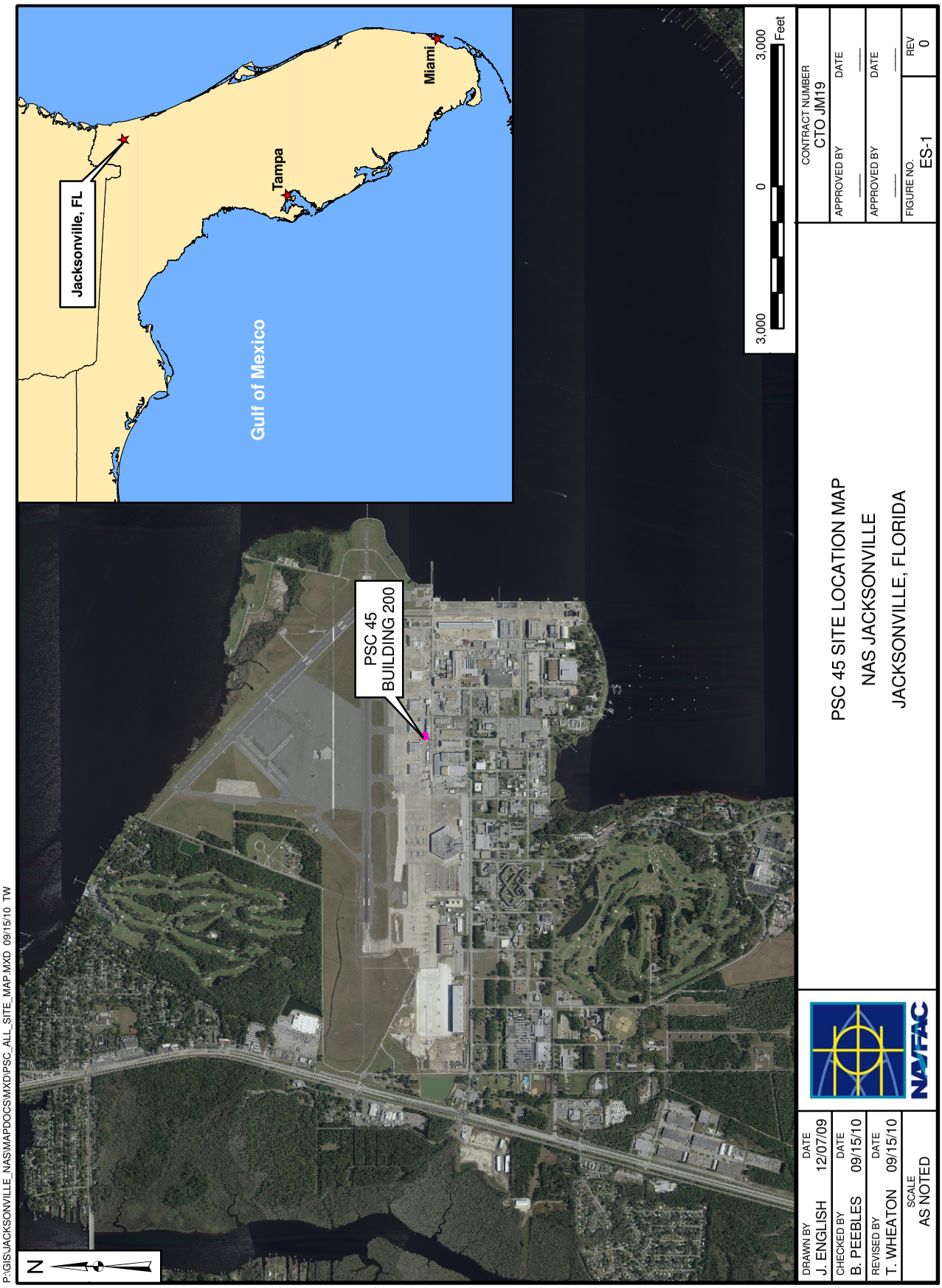
The primary objective of the SA was to determine if contamination exceeding project action limits (PALs) exist for soil and groundwater at PSC 45. If this condition was found to exist, then a Remedial Investigation (RI) would be implemented. If contamination exceeding the SA PALs was not found, then no further action would be warranted. The SA was not designed to characterize the nature or extent of the contamination in either soil or groundwater.

The soil and groundwater samples associated with the SA sampling event of August 2009 were found to contain multiple analytes at concentrations greater than the SA PALs. Unless land use patterns change, there is only limited potential for exposure to construction workers and there is no habitat for ecological receptors at PSC 45. Thus PSC 45 presents a possible risk to construction workers and no unacceptable risk to ecological receptors. However, it is reported that groundwater may be intercepted by storm sewers and these storm sewers discharge into the St. Johns River. Therefore, there is a potential for ecological receptors in the St. Johns River to be exposed to PSC 45 contaminants. This potential risk to ecological receptors is fully addressed under the current scope of investigation at Operational Unit (OU) 3 and the similarity of conditions between PSC 45 and OU 3 (both hydrological and contaminant type) and proximity of PSC 45 to OU 3 obviate the need to evaluate risk to ecological receptors solely from exposure to PSC 45 contaminants that may discharge into the St. Johns River via the storm sewer.

The objective of the RI is to determine the nature and extent of contamination and conduct a quantitative human health risk assessment (HHRA) and a Screening Level Ecological Risk Assessment (SLERA). The results of the HHRA and the SLERA will be used for the basis of a Feasibility Study (FS).

During the RI, surface soil, subsurface soil, and groundwater samples will be analyzed for the following groups of target analytes: VOCs, SVOCs, PCBs, TRPH, and metals. The data associated with the SA were used to help determine the locations of these soil and groundwater samples. Subsurface soil samples will be collected from the immediate area associated with the former Building 200 Wash Rack Disposal Pit. Surface soil samples will be collected in connection to the oil/water separator and the pit area, as the data from the SA indicated contaminants are present in this area. Most of the groundwater samples will be collected from locations that are 300 to 400 feet downgradient of the former Building 200 Wash Rack Disposal Pit, as the data from the SA indicated that the heaviest concentrations of contaminants are in this area. All surface soil, subsurface soil, and groundwater samples will be analyzed by analytical methods with sufficient sensitivity to ensure that the results can be used in the risk assessments.

Resulting RI data, which will be collected from carefully selected sampling locations, will be analyzed to establish the nature and extent of contamination and/or to estimate risks in accordance with United States Environmental Protection Agency Risk Assessment Guidance for Superfund sites.


Jacksonville, FL
Tampa
Miami
Gulf of Mexico
PSC 45
BUILDING 200
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PSC 45 SITE LOCATION MAP
NAS JACKSONVILLE
JACKSONVILLE, FLORIDA
DRAWN BY J. ENGLISH DATE 12/07/09
CHECKED BY B. PEEBLES DATE 09/15/10
REVISED BY T. WHEATON DATE 09/15/10
SCALE AS NOTED
NAVFAC
CONTRACT NUMBER CTO JM19
APPROVED BY DATE
APPROVED BY DATE
FIGURE NO. ES-1
REV 0

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SAP Worksheets **Page**

APPENDICES

FIGURES

NUMBER **PAGE**

TABLE

ACRONYMS AND ABBREVIATIONS

%D	Percent difference/drift
%R	Percent recovery
%RSD	Percent Relative Standard Deviation
°C	Degree Celsius
AES	Atomic Emissions Spectroscopy
BAP	Benzo(a)pyrene
BFB	Bromofluorobenzene
bgs	Below ground surface
CAS	Chemical Abstracts Service
CCB	Continuing Calibration Blank
CCC	Calibration Check Compound
CCV	Continuing Calibration Verification
CERCLA	Comprehensive Environmental Response, Compensation, and Liability Act of 1980
CFR	Code of Federal Regulations
CLEAN	Comprehensive Long-term Environmental Action Navy
CLLE	Continuous liquid to liquid extraction
CLP	Contract Laboratory Program
COPC	Chemical of Potential Concern
CTO	Contract Task Order
CVAA	Cold Vapor Atomic Absorption
DFTPP	Decaflurotriphenyl-phosphine
DoD	Department of Defense
DPT	Direct-push technology
DQI	Data Quality Indicator
DQO	Data Quality Objective
DVM	Data Validation Manager
EDD	Electronic data deliverable
ELAP	Environmental Laboratory Accreditation Program
EPC	Exposure Point Concentrations
EU	Exposure Unit
Ext.	Extension
F.A.C.	Florida Administrative Code
FDEP	Florida Department of Environmental Protection
FID	Flame ionization detector

ACRONYMS AND ABBREVIATIONS (CONTINUED)

FL-PRO	Florida Residual Petroleum Organics Method
FOL	Field Operations Leader
FS	Feasibility Study
FTMR	Field Task Modification Request
g	Gram
GC/ECD	Gas Chromatography/Electron Capture Detector
GC/FID	Gas Chromatography/Flame Ionization Detector
GC/MS	Gas Chromatography/Mass Spectrometer
GCTL	Groundwater Cleanup Target Level
GPS	Global Positioning System
HASP	Health and Safety Plan
HAZWOPER	Hazardous Waste Operations and Emergency Response
HCl	Hydrochloric Acid
HDOP	Horizontal Dilution of Precision
HHRA	Human Health Risk Assessment
HSM	Health and Safety Manager
ICAL	Initial Calibration
ICB	Initial Calibration Blank
ICP	Inductively Coupled Plasma
ICV	Initial Calibration Verification
ICS	Interference Check Standard
IDW	Investigation-derived waste
IR	Installation Restoration
IRP	Installation Restoration Program
IS	Internal Standard
Katahdin	Katahdin Analytical Services, Inc.
L	Liter
LCS	Laboratory Control Sample
LCSD	Laboratory Control Sample Duplicate
LOD	Limit of Detection
LOQ	Limit of Quantitation
LUC	Land Use Control
MDL	Method detection limit
mg/kg	Milligrams per kilogram
mg/L	Milligrams per liter

ACRONYMS AND ABBREVIATIONS (CONTINUED)

mL	Milliliter
MPC	Measurement Performance Criterion
MS	Matrix Spike
MSD	Matrix Spike Duplicate
NA	Not Applicable
NAS	Naval Air Station
NAVFAC SE	Naval Facilities Engineering Command Southeast
NC	No criteria
NELAP	National Environmental Laboratory Accreditation Program
NFA	No Further Action
NIRIS	Naval Installation Restoration Information Solution
NTU	Nephelometric Turbidity Unit
ORP	Oxidation Reduction Potential
OSHA	Occupational Safety and Health Administration
OU	Operable Unit
oz	Ounce
PAH	Polynuclear aromatic hydrocarbon
PAL	Project Action Limit
PCB	Polychlorinated Biphenyls
PDF	Portable Document Format
PID	Photoionization detector
PM	Project Manager
POC	Point of Contact
PPE	Personal Protective Equipment
PQO	Project Quality Objective
PSC	Potential Source of Contamination
PSL	Project Screening Level
PVC	Polyvinyl chloride
PWD	Public Works Department
QA	Quality Assurance
QAM	Quality Assurance Manager
QAO	Quality Assurance Officer
QAPP	Quality Assurance Project Plan
QC	Quality Control
QL	Quantitation Limit

ACRONYMS AND ABBREVIATIONS (CONTINUED)

r	Correlation Coefficient
r2	Coefficient of Determination
RAC	Remedial Action Contractor
RBRSL	Risk-Based Regional Screening Level
RF	Response Factor
RI	Remedial Investigation
RPD	Relative Percent Difference
RPM	Remedial Project Manager
R-RSL	Residential Regional Screening Level
RRT	Relative Retention Time
RSD	Relative Standard Deviation
RT	Retention Time
SA	Site Assessment
SAP	Sampling and Analysis Plan
SCTL	Soil Cleanup Target Level
SD	Serial Dilution
SDG	Sample Delivery Group
SIM	Selected Ion Monitoring
SLERA	Screening Level Ecological Risk Assessment
SOP	Standard Operating Procedure
SPCC	System Performance Check Compound
SQL	Structured Query Language
SSO	Site Safety Officer
SVOC	Semivolatile Organic Compound
TBD	To Be Determined
TCLP	Toxicity Characteristic Leaching Procedure
TEF	Toxic Equivalency Factor
Tetra Tech	Tetra Tech NUS, Inc.
TPH	Total Petroleum Hydrocarbons
TRPH	Total Recoverable Petroleum Hydrocarbons
UCL	Upper Confidence Limit
UFP	Uniform Federal Policy
µg/L	Micrograms per liter
USEPA	United States Environmental Protection Agency
VOC	Volatile organic compound

SAP Worksheet #2 -- SAP Identifying Information

(UFP-QAPP Manual Section 2.2.4)

Site Name/Number:	Naval Air Station (NAS) Jacksonville, Florida
Operable Units:	Potential Source of Contamination (PSC) 45
Contractor Name:	Tetra Tech, NUS Inc. (Tetra Tech)
Contract Number:	N62470-08-D-1001
Contract Title:	Comprehensive Long-term Environmental Action Navy (CLEAN)
Work Assignment Number:	Contract Task Order (CTO) JM19

1. This Sampling and Analysis Plan (SAP) was prepared in accordance with the requirements of the *Uniform Federal Policy for Quality Assurance Project Plans (UFP-QAPP)* (USEPA, 2005) and the United States Environmental Protection Agency (USEPA) *Guidance for Quality Assurance Project Plans, QA/G-5, QAMS* (USEPA, 2002).
2. Identify regulatory program: Comprehensive Environmental Response, Compensation, and Liability Act of 1980 (CERCLA).
3. This SAP is a project-specific SAP.
4. List dates of scoping sessions that were held:

Scoping Session	Date
Meeting No. 1 – Site Walk	October 03, 2008
Meeting No. 2 – Site Walk	October 08, 2008
Meeting No. 3 – Partnering/Data Quality Objective (DQO) Scoping Meeting	October 21-22, 2008
Meeting No. 4 – DQO Scoping Meeting	November 19, 2008
Meeting No. 5 – Planning .	April 20-21, 2010 .

5. List dates and titles of any SAP documents written for previous site work that are relevant to the current investigation.

Title	Date
Potential Source of Contamination 45, No Further Response Action Planned or Further Remedial Action Decision Report. Tetra Tech, 2004	Internal document dated August 27, 1999 identified in Tetra Tech, 2004
Sampling and Analysis Plan (Field Sampling Plan and Quality Assurance Project Plan) Site Assessment Activities Potential Source of Contamination (PSC 45) Building 200 Wash Rack. Tetra Tech, 2009	April 2009

6. List organizational partners (stakeholders) and connection with lead organization:

USEPA Region 4 – Regulatory Oversight
Florida Department of Environment Protection (FDEP) – Regulatory Oversight
NAS Jacksonville – Property Owner

7. Lead organization

Naval Facilities Engineering Command Southeast (NAVFAC SE)

- If any required SAP elements or required information are not applicable to the project or are provided elsewhere, then note the omitted SAP elements and provide an explanation for their exclusion below:

Not Applicable (NA), as there are no exclusions.

SAP Worksheet #3 – Distribution List
(UFP-QAPP Manual Section 2.3.1)

Name of SAP Recipients	Title/Role	Organization	Telephone Number	E-Mail Address or Mailing Address
Adrienne Wilson	Navy Remedial Project Manager (RPM)/ Manages Project Activities for the Navy	Department of the Navy Naval Facilities Southeast Code OPDE3/AW Attn: Ajax Street, Bldg 135N P.O. Box 30A Jacksonville, FL 32212-0030	(904) 542-6160	Adrienne.Wilson@navy.mil
Tim Curtin	Installation Restoration Program (IRP) Manager/ NAS Jacksonville Point of Contact (POC)	NAS Jacksonville Building 1, Code 064TC NASJAX /Yorktown/Langley Jacksonville, FL 32212	(904) 542-4228	Tim.L.Curtin@navy.mil
To Be Determined (TBD)	NAVFAC Quality Assurance Officer (QAO)/ Government Chemist	TBD	TBD	TBD
David Grabka	FDEP RPM/ Provides Regulator Input	FDEP 2600 Blair Stone Road, MS 4535 Tallahassee, FL 32399-2400	(850) 245-8997	david.grabka@dep.state.fl.us
Peter Dao	USEPA RPM/ Provides Regulator Input	USEPA Region 4 Atlanta Federal Center 61 Forsyth Street, SW Atlanta, GA 30303-8960	(404) 562-8508	dao.peter@epa.gov
Bonnie Capito	Librarian and Records Manager	Naval Facilities Engineering Command Code 1832 5 l 0 Gilbert Street. Norfolk, VA 23511-2699	757-322-4785	Bonnie.Capito@navy.mil
John Trepanowski (copy of cover letter only)	Tetra Tech Program Manager / Manages Navy Initiatives and CLEAN Program	Tetra Tech 234 Mall Boulevard Suite 260 King of Prussia, PA 19406	(610) 382-1532	john.trepanowski@tetratech.com

Name of SAP Recipients	Title/Role	Organization	Telephone Number	E-Mail Address or Mailing Address
Garth Glenn (copy of cover letter only)	Deputy Program Manager/ Manages CLEAN Program Activities	Tetra Tech 5700 Lake Wright Drive Suite 309 Norfolk, VA 23502	(757) 461-3926	garth.glenn@tetratech.com
Mark Peterson	Project Manager (PM)/ Manages Project Activities	Tetra Tech 8640 Philips Hwy, Suite 16 Jacksonville, FL 32256	(904) 730-4669 Extension (Ext.) 213	mark.peterson@tetratech.com
Alan Pate	Field Operations Leader (FOL) / Site Safety Officer (SSO)/ Manages Field Operation and Site Safety Issues	Tetra Tech 8640 Philips Hwy, Suite 16 Jacksonville, FL 32256	(904) 730-4669 Ext. 214	alan.pate@tetratech.com
Tom Johnston, PhD (electronic copy only)	Quality Assurance Manager (QAM)/ Manages Corporate Quality Assurance (QA) Program and Implementation	Tetra Tech 661 Andersen Drive Foster Plaza 7 Pittsburgh, PA 15220	(412) 921-8615	tom.johnston@tetratech.com
Matt Kraus	Project Chemist/ Provides Coordination with Laboratory	Tetra Tech 661 Andersen Drive Foster Plaza 7 Pittsburgh, PA 15220	(412) 921-8729	matthew.kraus@tetratech.com
Peggy Churchill (electronic copy only)	Environmental Scientist/ Provides DQO and SAP Support	Tetra Tech 11 Riverside Drive, Suite 206 Cocoa, FL 32922	(321) 636-6470	peggy.churchill@tetratech.com
Matt Soltis [Health and Safety Plan (HASP) only]	Health and Safety Manager (HSM)/ Manages Corporate Health and Safety Program	Tetra Tech 661 Andersen Drive Foster Plaza 7 Pittsburgh, PA 15220	(412) 921-8912	matt.soltis@tetratech.com
Joseph Samchuck (electronic copy only)	Data Validation Manager (DVM)/ Manages Data Validation	Tetra Tech 661 Andersen Drive Foster Plaza 7 Pittsburgh, PA 15220	(412) 921-8510	joseph.samchuck@tetratech.com

Name of SAP Recipients	Title/Role	Organization	Telephone Number	E-Mail Address or Mailing Address
Julie Johnson	Administrative Project Assistant (NAS Jacksonville Administrative Record)	Tetra Tech 8640 Philips Hwy, Suite 16 Jacksonville, FL 32256	(904) 739-4669 Ext. 224	julie.johnson@tetratech.com
Kelly Perkins (electronic copy only)	Laboratory PM/ Representative for Laboratory and Analytical Issues	Katahdin Analytical Services, Inc. (Katahdin) 600 Technology Way Scarborough, ME 04070	(207) 874-2400 Ext. 17	kperkins@katahdinlab.com
Well Installation Subcontractor (TBD) (electronic copy only)	Well Installation Subcontractor PM/ Provides Drilling Services	TBD	TBD	TBD

SAP Worksheet #4 – Project Personnel Sign-Off Sheet
(UFP-QAPP Manual Section 2.3.2)

Certification that project personnel have read the text will be obtained by one of the following methods as applicable:

1. In the case of regulatory agency personnel with oversight authority, approval letters or e-mails will constitute verification that applicable sections of the SAP have been reviewed. Copies of regulatory agency approval letters / e-mails will be retained in the project files and are listed in Worksheet #29 as project records.

2. E-mails will be sent to the Navy, Tetra Tech, and subcontractor project personnel who will be requested to verify by e-mail that they have read the applicable SAP / sections and the date on which they were reviewed. Copies of the verification e-mail will be included in the project files and is identified in Worksheet #29.

   A copy of the signed Worksheet #4 will be retained in the project files and is identified as a project document in Worksheet #29.

Name	Organization/Title/Role	Telephone Number	Signature/E-Mail Receipt	SAP Section Reviewed	Date SAP Read
Navy and Regulator Partnering Team Personnel					
Adrienne Wilson	Navy/ RPM/ Manages Project Activities for the Navy	(904) 542-6160	See Worksheet #1 for signature	All	
Tim Curtin	Navy/ IRP Manager/ NAS Jacksonville POC	(904) 542-4228		All	
David Grabka	FDEP/ RPM/ Provides Regulator Input	(850) 245-8997	See Worksheet #1 for signature	All	
Peter Dao	USEPA Region 4/ RPM/ Provides Regulator Input	(404) 562-8508	See Worksheet #1 for signature	All	

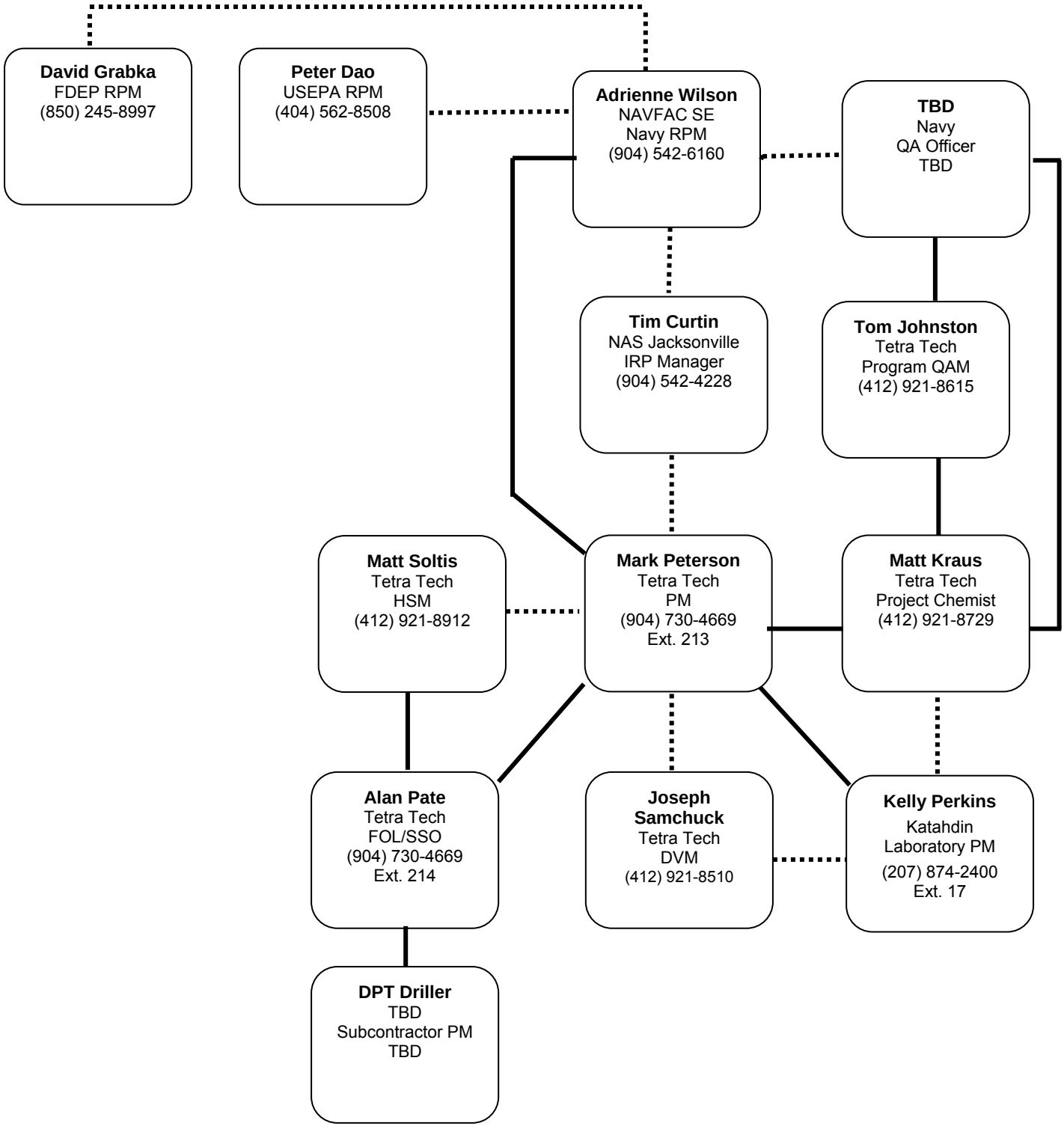
Name	Organization/Title/Role	Telephone Number	Signature/E-Mail Receipt	SAP Section Reviewed	Date SAP Read
Tetra Tech Partnering Team Personnel					
Mark Peterson	Tetra Tech/ PM/ Manages Project Activities	(904) 730-4669 Ext. 213	See Worksheet #1 for signature	All	
Alan Pate	Tetra Tech/ FOL/SSO/ Manages Field Operation and Site Safety Issues	(904) 730-4669 Ext. 214		All	
Tom Johnston	Tetra Tech/ QAM/ Manages NAVFAC SE Contract QA Program and Implementation	(412) 921-8615	See Worksheet #1 for signature	All	
Matt Soltis	Tetra Tech/ HSM/ Manages Corporate Health and Safety Program	(412) 921-8912	See HASP for signature	HASP	
Peggy Churchill	Tetra Tech/ Environmental Scientist/ Provides DQO and SAP Support	(321) 636-6470		All	
Matt Kraus	Tetra Tech/ Project Chemist/ Provides Coordination with Laboratory	(803) 641-4944		All	
Joseph Samchuck	Tetra Tech/ DVM/ Manages Data Validation	(412) 921-8510		Worksheets #12, #14, #15, #19, #20, #23-28, #30, and #34-37	

Name	Organization/Title/Role	Telephone Number	Signature/E-Mail Receipt	SAP Section Reviewed	Date SAP Read
Subcontractor Personnel					
Kelly Perkins	Katahdin/ Laboratory PM/ Representative for Laboratory and Analytical Issues	(207) 874-2400 Ext. 17		Worksheets #6, #12, #14, #15, #19, #23-28, #30, and #34-36	
TBD	Well Installation Subcontractor PM/ Provides Drilling Services	TBD		Worksheets #6, #14, #17, and Figures	

SAP Worksheet #5 – Project Organizational Chart

(UFP-QAPP Manual Section 2.4.1)

Lines of Authority ——— Lines of Communication



SAP Worksheet #6 – Communication Pathways
(UFP-QAPP Manual Section 2.4.2)

Communication Drivers	Responsible Affiliation	Name	Phone Number and/or E-Mail	Procedure (timing, pathway to & from, etc.)
SAP amendments	Tetra Tech FOL/SSO Tetra Tech PM Navy RPM	Alan Pate Mark Peterson Adrienne Wilson	(904) 730-4669 Ext. 214 (904) 730-4669 Ext. 213 (904) 542-6160	The Tetra Tech FOL will verbally inform the Tetra Tech PM within 24 hours of realizing a need for an amendment. The Tetra Tech PM will document the proposed changes via a Field Task Modification Request (FTMR) form within 5 days and send the Navy RPM a concurrence letter within 7 days of identifying the need for change. SAP amendments will be submitted by the Tetra Tech PM to the Navy RPM for review and approval. The Navy RPM will notify the regulators by mail of changes to the SAP. The Tetra Tech PM will send scope changes to the Partnering Team via e-mail within 1 business day.
Changes in schedule	Tetra Tech PM Navy RPM NAS Jacksonville POC	Mark Peterson Adrienne Wilson Tim Curtin	(904) 730-4669 Ext. 213 (904) 542-6160 (904) 542-4228	The Tetra Tech PM will verbally inform the Navy RPM and the NAS Jacksonville RPM on the day that schedule change is known and document via schedule impact letter within 1 business day of when impact is realized.

Communication Drivers	Responsible Affiliation	Name	Phone Number and/or E-Mail	Procedure (timing, pathway to & from, etc.)
Issues that lead to changes in the scope of work. This scope change includes other than field-change drivers and such change may require SAP amendments.	Tetra Tech FOL/SSO Tetra Tech PM Navy RPM NAS Jacksonville POC	Alan Pate Mark Peterson Adrienne Wilson Tim Curtin	(904) 730-4669 Ext. 214 (904) 730-4669 Ext. 213 (904) 542-6160 (904) 542-4228	The Tetra Tech FOL will verbally inform the Tetra Tech PM on the day that the issue is discovered. The Tetra Tech PM will inform the Navy RPM and the NAS Jacksonville IRP Manager (verbally or via e-mail) within 1 business day of discovery. The Navy RPM will issue scope change (verbally or via e-mail), if the change is significant enough to warrant a scope change. The scope change is to be implemented before further work is executed. The Tetra Tech PM will document the change via an FTMR form within 2 days of identifying the need for change and will obtain required approvals within 5 days of initiating the form.
Recommendation to stop work and initiate work upon corrective action	Tetra Tech FOL/SSO Tetra Tech PM Tetra Tech QAM Tetra Tech HSM Tetra Tech Project Chemist Navy RPM NAS Jacksonville POC	Alan Pate Mark Peterson Tom Johnston Matt Soltis Matt Kraus Adrienne Wilson Tim Curtin	(904) 730-4669 Ext. 214 (904) 730-4669 Ext. 213 (412) 921-8615 (412) 921-8912 (412) 921-8729 (904) 542-6160 (904) 542-4228	If Tetra Tech is the responsible party for a stop work command, the Tetra Tech FOL will inform on-site personnel, subcontractor(s), the NAS Jacksonville POC, and the identified Partnering Team members within 1 hour (verbally or by e-mail). If a subcontractor is the responsible party, the subcontractor PM must inform the Tetra Tech FOL within 15 minutes, and the Tetra Tech FOL will then follow the procedure listed above.

Communication Drivers	Responsible Affiliation	Name	Phone Number and/or E-Mail	Procedure (timing, pathway to & from, etc.)
Corrective action for field, analytical/laboratory, or reporting program	Tetra Tech QAM Tetra Tech PM Navy RPM Navy QAO	Tom Johnston Mark Peterson Adrienne Wilson TBD	(412) 921-8615 (904) 730-4669 Ext. 213 (904) 542-6160 TBD	The Tetra Tech QAM will notify the Tetra Tech PM verbally or by e-mail within 1 business day that the corrective action has been completed. The Tetra Tech QAM will notify the Navy QAO and the Tetra Tech PM within 1 business day that the corrective action on an analytical/laboratory issue has been completed. The Tetra Tech PM will then notify the Navy RPM (verbally or by e-mail) within 1 business day.
Field data quality issues	Tetra Tech FOL/SSO Tetra Tech PM	Alan Pate Mark Peterson	(904) 730-4669 Ext. 214 (904) 730-4669 Ext. 213	The Tetra Tech FOL will inform the Tetra Tech PM (verbally or by e-mail) on the same day that a field data quality issue is discovered.
Analytical data quality issues	Laboratory PM Tetra Tech Project Chemist Navy RPM Tetra Tech PM Tetra Tech QAM	Kelly Perkins Matt Kraus Adrienne Wilson Mark Peterson Tom Johnston	(207) 874-2400 Ext. 17 (412) 921-8729 (904) 542-6160 (904) 730-4669 Ext. 213 (412) 921-8615	The Laboratory PM will notify (verbally or via e-mail) the Tetra Tech Project Chemist within 1 business day of when an issue related to laboratory data is discovered. The Tetra Tech Project Chemist will notify (verbally or via e-mail) the data validation staff and the Tetra Tech PM within 1 business day. The Tetra Tech PM will notify the Navy RPM and the Tetra Tech QAM (verbally or via e-mail) of significant data quality issues within 1 business day of resolution. The Tetra Tech QAM will notify the Navy QAO within 2 business days of resolution.

SAP Worksheet #7 – Personnel Responsibilities and Qualifications Table
(UFP-QAPP Manual Section 2.4.3)

Name	Title/Role	Organizational Affiliation	Responsibilities
Adrienne Wilson	Navy RPM/ Manages project activities for the Navy	NAVFAC SE	Oversees project implementation including scoping, data review, and evaluation.
Tim Curtin	IRP Manager/ Manages daily site activities related to this project	NAS Jacksonville	Oversees site activities and participates in scoping, data review, evaluation, and reviews the SAP.
David Grabka	RPM/ Provides regulator input	FDEP	Participates in scoping, data review, evaluation, and approves the SAP.
Peter Dao	RPM/ Provides regulator input	USEPA Region 4	Participates in scoping, data review, evaluation, and approves the SAP.
Mark Peterson	PM/ Manages project on a daily basis	Tetra Tech	Oversees project and manages financial, schedule, and technical day-to-day activities of the project.
Alan Pate	FOL/SSO Manages field operations and oversees site activities to ensure safety requirements are met	Tetra Tech	As FOL, supervises, coordinates, and performs field sampling activities. As the SSO, is responsible for on-site project-specific health and safety training and monitoring site conditions. Details of these responsibilities are presented in the HASP.
Tom Johnston	QAM/ Oversees program and project QA activities	Tetra Tech	Reviews the SAP and ensures quality aspects of the CLEAN program are implemented, documented, and maintained.
Matt Soltis	HSM/ Oversees health and safety activities	Tetra Tech	Oversees CLEAN Program Health and Safety Program.
Matt Kraus	Project Chemist/ Conducts data validation and reporting	Tetra Tech	Participates in project scoping, prepares laboratory scopes of work, and coordinates laboratory-related functions with laboratory. Oversees data quality reviews and QA of data validation deliverables.
Joseph Samchuck	DVM/ Oversees data validation activities	Tetra Tech	Manages data validation activities within Tetra Tech, including ensuring QA of data validation deliverables, providing technical advice on data usability, and coordinating and maintaining the data validation review schedule.

Name	Title/Role	Organizational Affiliation	Responsibilities
Kelly Perkins	Laboratory PM/ Representative for Laboratory and Analytical Issues	Katahdin	Coordinates analyses with laboratory chemists, ensures that scope of work is followed, provides QA of data packages, and communicates with Tetra Tech project staff.
TBD	Well Installation Subcontractor	TBD	Performs DPT soil borings according to scope of work.
TBD	Utility Locator	TBD	Performs utility location.

SAP Worksheet #8 – Special Personnel Training Requirements Table

(UFP-QAPP Manual Section 2.4.4)

Each site worker will be required to have completed appropriate Hazardous Waste Operations and Emergency Response (HAZWOPER) training specified in Occupational Safety and Health Administration (OSHA) 29 Code of Federal Regulations (CFR) 1910.120 (e). Project-specific safety requirements are addressed in greater detail in the site-specific HASP.

SAP Worksheet #9 – Project Scoping Session Participants Sheet

(UFP-QAPP Manual Section 2.5.1)

PARTNERING TEAM MEETING
Date of Session: April 20-21, 2010
PSC 45 Scoping Session Purpose: Planning

Name	Title	Affiliation	Phone Number	E-Mail Address	Project Role
Adrienne Wilson	Navy RPM	NAVFAC Southeast	(904) 542-6160	Adrienne.Wilson@ navy.mil	NAVFAC SE RPM
Tim Curtin	IRP Manager	NAS Jacksonville	(904) 542-4228	Tim.L.Curtin@navy.mil	IRP Manager
David Grabka	State RPM	FDEP	(850) 245-8997	David.Grabka@ dep.state.fl.us	Regulatory
Peter Dao	USEPA RPM	USEPA Region IV	(404) 562-8508	dao.peter@ epa.gov	Regulatory
Hal Davis	Hydrogeologist	USGS	(850) 942-9500	hdavis@usgs.gov	Technical support
Mark Peterson	PM	Tetra Tech	(904) 636-6125	mark.peterson@ tetratech.com	Project Management
Casey Hudson	PM	CH2M Hill Constructors, Inc. (CH2M Hill)	(770) 604-9182	casey.hudson@ ch2m.com	Technical input RAC Contractor
Eric Davis	Remedial Action Contract (RAC) Contractor	CH2M Hill	(678) 530-4085	Eric.Davis@ch2m.com	RAC Contractor
Julie Johnson	Administrative Project Assistant	Tetra Tech	(904) 730-4669 Ext. 224	Julie.Johnson@tetratech.com	Scribe
Guests					
Name	**Title**	**Affiliation**	**Phone Number**	**E-Mail Address**	**Project Role**
Mike Maughon	Senior Environmental Engineer	Tetra Tech	(843) 886-4547	Mike.Maughon@tetratech.com	Technical Support: Assessment, Regulatory Compliance, and Remediation
Alan Pate	FOL	Tetra Tech	(904) 730-4669 Ext. 214	Alan.Pate@tetratech.com	Project Support

Comments/Decisions: The approach to the SA was fully developed in the April 2009 Site Assessment Activities Sampling Analysis Plan (Field Sampling Plan And Quality Assurance Project Plan) for PSC 45, Building 200 Wash Rack.

April 2010 DQO Session

Action Items: Develop a SAP for RI fieldwork at PSC 45 as soil and groundwater samples associated with the

PARTNERING TEAM MEETING
Date of Session: April 20-21, 2010
PSC 45 Scoping Session Purpose: Planning

SA contained analytes at concentrations that were above the SA specific Project Action Limits (PALs).
Notes: (a) The SA analytical results are summarized in Appendix A of this SAP; (b) the term Project Action Limits or PALs was used during the SA as these action limits were used to trigger a major action; i.e., the decision to conduct the RI or proceed to a no further action (NFA) request; (c) the term Project Screening Level (PSL) is also used in this RI SAP as the comparison of site data to the PSL will be used to select chemicals of potential concern (COPCs) and help define the extent of contamination.

Consensus Decisions: PSC 45 – Building 200 Wash Rack -- Team reached consensus to utilize the same methods, PALs, and procedures covered in the UFP SAP for the SA at this site to complete the RI. Team agreed that the problem statement and decision rules should be updated to reflect investigation and delineation of the sources of contamination.

SAP Worksheet #10 - Conceptual Site Model
(UFP-QAPP Manual Section 2.5.2)

10.1 INTRODUCTION

NAS Jacksonville was commissioned in October 1940 to provide facilities for air operations and pilot training and a Navy Aviation Trades School for ground crewmen. NAS Jacksonville is located in Duval County, Florida on the western bank of the St. Johns River (Figure 10-1). This facility is approximately 3,800 acres in size and its current mission is to provide facilities and support for the operation and maintenance of Naval weapons and aircraft.

The main portion of NAS Jacksonville is bordered to the north by the Timuquana Country Club, to the east and northeast by the St. Johns River, to the south by a residential area, and to the west by Highway 17, with Westside Regional Park and commercial developments. The facility is located approximately 24 miles inland from the Atlantic Ocean.

NAS Jacksonville is home to Patrol Squadron Thirty (VP-30), the Navy's largest aviation squadron and the only P-3 Orion Fleet Replacement Squadron that prepares and trains U.S. and foreign pilots, air crew, and maintenance personnel for further operational assignments. Support facilities include an airfield for pilot training, a maintenance depot employing more than 150 different trade skills capable of performing maintenance as basic as changing a tire to intricate micro-electronics or total engine disassembly, a Naval hospital, a Fleet Industrial Supply Center, and a Navy Family Service Center.

Work in support of the base mission includes fuel storage and transportation systems and the overhaul, intermediate maintenance, and repair of aircraft and engines. Maintenance activities at NAS Jacksonville over the years generated a variety of waste materials, of which some were disposed of on the base. These include materials resulting from construction activities; municipal solid waste and municipal wastewater treatment plant sludge; and miscellaneous industrial wastes including waste oils or solvents, paints, and spilled fuels. Current disposal practices are regularly surveyed for conformity to local, state, and federal regulations.

This SAP governs soil and groundwater sampling and analyses and related RI activities at PSC 45 (the former Building 200 Wash Rack Disposal Pit) located on the NAS Jacksonville facility (Figure 10-2).

10.2 SITE DESCRIPTION

Building 200 is a ground support equipment facility and is located on the northern industrialized part of NAS Jacksonville near the flight line (Figure 10-3). A covered wash rack with a floor drain leading to an

oil/water separator (located beneath wash rack) was located on the northwest corner of the building. An overflow pipe from the oil/water separator was connected to a cylindrical concrete disposal pit located approximately 2 feet east of the covered wash rack area. The pit was a French drain design that leached directly into the subsurface soil. The pit was gravel filled with an earthen bottom and a concrete lid approximately 4 feet in diameter. A small grassy area surrounded the former disposal pit and a paved parking lot is located north of this grassy area. The Building 200 Wash Rack Disposal Pit was identified as a PSC 45 by NAS Jacksonville in 1991.

In the past, ground support equipment was cleaned in the wash rack and while in the wash rack, solvents were used to strip paint off the equipment. For an unknown period of time (up to 1991), the disposal pit received overflow from an oil-water separator in the wash rack. According to a Hazardous Waste Manager for Building 200, no maintenance was ever done on the oil/water separator (Tetra Tech, 2004). The disposal pit was discovered in 1991 during plumbing repair work at Building 200. After the pit was discovered, the connection from the oil/water separator to the pit was plugged and waste from the pit was removed and disposed of as hazardous waste.

A new oil/water separator was installed within the excavation area. The old oil/water separator is still operational in the wash rack room; however, to further safeguard against the accidental release of oil and solvents, effluent from the old separator is directed through plumbing to the new separator before going directly to the sanitary sewer.

The stratigraphy at PSC 45 has not been fully determined. However, PSC 45 is approximately 400 feet northwest of Operational Unit (OU) 3; therefore, the stratigraphy at PSC 45 is assumed to be similar to the stratigraphy at OU 3, which consists of silty to clayey sands interbedded with layers of clay and sandy clay. In the northern half of OU 3, a clay layer separates the shallow and intermediate zones of the surficial aquifer and may be thinned and discontinuous (Tetra Tech, 2010). Groundwater migration above the clay layer in the northern part of OU 3 is generally to the east towards the St. Johns River, but is affected by leaky storm sewers with inverts located beneath the water table. Below the clay layer in the northern portion of OU 3, groundwater flows east toward the St. Johns River and then upward along the saltwater interface upgradient of the river. Groundwater flow associated with PSC 45 is expected to mirror what is seen at OU 3. That is, groundwater generally flows east towards the St. Johns River. However, storm sewer inverts are located beneath the water table and may intercept groundwater which is then conveyed to the St. Johns River (Tetra Tech, 2004).

10.3 PREVIOUS INVESTIGATIONS

A liquid sample was collected from the disposal pit in July 1991, probably before waste was removed from the pit. The gross components of the sample were water, paint chips, paint stripper, and oil. A

methylene chloride concentration of 1,800 milligrams per liter (mg/L) and a phenol concentration of 285 mg/L were measured in the sample. In 1994, a sludge sample was collected from the disposal pit and analyzed for oil and grease only; the result was 7.8 mg/L. In 1998, the Wash Rack Disposal Pit, the liquid and solids within the pit, and the soil surrounding and underlying the pit were removed. Pre-disposal samples collected from the soil, liquid, and solids were analyzed using the Toxicity Characteristic Leaching Procedure (TCLP) for waste characterization and analyte concentrations were less than the applicable TCLP regulatory limits. No documentation of post-excavation sampling around the former pit has been found (Tetra Tech, 2004).

In August 2009, as part of the SA for PSC 45, Tetra Tech collected eight soil samples from four soil borings in the area of the former disposal pit (See Figure A-1 in Appendix A). Four groundwater samples were collected from one of the borings associated with the soil samples. Forty additional groundwater samples were collected from 10 borings advanced using direct push technology (DPT), along the east, north, and west sides of Building 200 (See Figure A-2 in Appendix A). Groundwater samples, from a total of 11 locations, were collected from four intervals: 12 to 16, 20 to 24, 40 to 44, and 60 to 64 feet below ground surface (bgs). The soil samples were analyzed for metals, volatile organic compounds (VOCs), semivolatile organic compounds (SVOCs) [including low level polynuclear aromatic hydrocarbons (PAHs)], total recoverable petroleum hydrocarbons (TRPH), and polychlorinated biphenyls (PCBs). Groundwater samples were analyzed for VOCs using a mobile laboratory.

The results of the SA confirmed that analyte concentrations, in excess of the SA-specific PALs (which the NAS Jacksonville Partnering Team agreed will be the RI-specific PALs) are present in soil and groundwater. A summary of detections by media and analyte group is presented in Table 10-1. A few VOCs were present in soil at concentrations greater than the RI PALs; however, PAHs appeared to be the primary human health risk driver in soil as a number of these compounds were detected at concentrations greater than the FDEP Soil Cleanup Target Levels (SCTLs) associated with direct exposure by commercial/industrial land use (Figure A-1 in Appendix A).

During the SA, groundwater was analyzed using a mobile laboratory for a reduced set of target analytes (see Table 10-1). Fewer analytes were detected in groundwater in the vicinity of the former Wash Rack Disposal Pit, and the concentrations of these analytes were generally less than concentrations in groundwater from 300 to 500 feet downgradient of the former Wash Rack Disposal Pit (Figure A-2 in Appendix A). The majority of analytes detected in groundwater were VOCs that are not usually associated with oil or petroleum hydrocarbons. Most of the analytes were detected in groundwater collected downgradient of the former Wash Rack Disposal Pit. The greatest frequencies of detection were in the 20 to 24 and the 40 to 44 feet bgs interval. No analytes were detected in the 60 to 64 feet bgs interval from the wells installed downgradient of the former Wash Rack Disposal Pit. At a DPT boring

installed at the former Wash Rack Disposal Pit, four analytes (1,2,4-trimethylbenzene, m+p-xylenes, o-xylene, and toluene) were detected in groundwater from the 60 to 64 feet bgs interval at concentrations below their PALs . Therefore the vertical extent of contamination does not extend below 64 feet bgs.

10.4 CONCEPTUAL SITE MODEL

The site conceptual model is presented in Figures 10-4 and 10-5. An oil water separator associated with the wash rack previously was piped to a disposal pit located near the northwest corner of Building 200. Effluent discharge entered the subsurface from the disposal pit prior to 1991 when the disposal pit was discovered and subsequently removed and replaced with an oil water separator piped to the sanitary sewer system.

The SA evaluated shallow soils in the vicinity of the plume and groundwater for the presence of potential contaminants of concern. A screening approach was employed where soil was evaluated for a wide range of potential contaminants of concern with testing conducted via a fixed-base laboratory and groundwater conditions were evaluated for a more limited set of analyses via a mobile laboratory. Based on the results of the SA, potential COCs were detected in shallow soils exceeding PALs. Contaminants detected in soils include metals, PCBs, VOCs, SVOCs and TRPH. Contaminants detected in groundwater included VOCs and SVOCs; however, it should be noted groundwater was tested for a limited parameter list that did not include metals, PCBs, or TRPH. The individual target analytes are presented in Worksheet #15.

The pipeline from the oil/water separator connected to the disposal pit was several feet below the ground surface. Shallow soil contamination detected in the SA is relatively minor and believed to be restricted to the near vicinity of the former disposal pit location. It is believed that contaminants bound in shallow soils near the former disposal pit location may serve as a continuing source for groundwater contamination. Contaminants can be leached from the subsurface soil and enter the groundwater through rainfall infiltration. The extent of PSC 45 contamination in soil and groundwater has not been completely defined (Figure 10-5). Furthermore, when the disposal pit was operational, groundwater may have been contaminated by direct discharge of liquids into the groundwater from the disposal pit. Based upon the hydrology associated with nearby OU 3, it is expected that groundwater associated with PSC 45 flows east toward the St. Johns River. Local topography decreases in elevation toward the east and/or southeast. Groundwater elevations typically mimic topography, thus groundwater is expected to flow toward the St. Johns River, which is approximately 3,000 feet east of PSC 45.

It is surmised that less mobile contaminants including metals and PCBs may be restricted to shallow soils in the vicinity of the former disposal pit and are not likely widely distributed in groundwater, while more mobile VOC contaminants may have migrated further from the disposal pit. SA data suggest that a VOC

groundwater plume is present at depths greater than 20 feet downgradient of the former disposal pit location at concentrations significantly higher than in the vicinity of the presumed source, indicating a potentially detached groundwater plume. This effect would be consistent with an older source area where discharges have been discontinued, as is the case with the former disposal pit.

Current land use in and around the Building 200 wash rack is industrial and will remain so for the foreseeable future. The current route of exposure, which is related to the types of activities that occur at an industrial site, is limited to dermal (direct) contact with soil, incidental ingestion, and inhalation by construction, maintenance or utility workers during excavation projects. Should a change in land use occur, then other human receptors may have the potential for exposure to contaminants. If land were transferred for unrestricted use, receptors could include future adult and child residents and recreationists.

The site itself does not contain the type of habitat that would be exploited by ecological receptors as PSC 45 is near the flight line, contains several buildings, several parking lots, is frequently maintained, and human activity in that area discourages the habitation by ecological receptors. Furthermore, the contaminants are considered to be bound in surface soil and ecological receptors are considered not to be exposed to subsurface soil; therefore, there is a lack of ecological receptors and exposure pathways at PSC 45 and the site likely presents no unacceptable risk to ecological receptors. Nonetheless, it is known that shallow groundwater in this area of NAS Jacksonville may be intercepted by storm sewers and these storm sewers discharge into the St. Johns River (Tetra Tech, 2004; see Figure 10-4 in Tetra Tech, 2010). Therefore, there is a potential for ecological receptors in the St. Johns River to be exposed to PSC 45-related groundwater contaminants. However, this potential risk to ecological receptors is fully addressed under the current scope of investigation at OU 3 (Tetra Tech, 2010) and the similarity of conditions between PSC 45 and OU 3 (both hydrological and contaminant type) and proximity of PSC 45 to OU 3 obviate the need to evaluate risk to ecological receptors from exposure to PSC 45 contaminants that may discharge into the St. Johns River via the storm sewer.

TABLE 10-1

ANALYTES WITH A DETECTED CONCENTRATION ABOVE THE REMEDIAL INVESTIGATION PALS[1] POTENTIAL SOURCE OF CONTAMINATION 45 NAS JACKSONVILLE JACKSONVILLE, FLORIDA

PARAMETER	Groundwater	Subsurface Soil	Surface Soil
METALS			
CADMIUM	NT[2]	●	●
CALCIUM	NT	●	●
COBALT	NT	●	●
IRON	NT	*[3]	●
MAGNESIUM	NT	*	●
MERCURY	NT	*	●
PCBs			
AROCLOR-1260	NT	●	●
SVOCs			
BAP EQUIVALENT[4]	NT	●	●
BENZO(A)ANTHRACENE	NT	●	●
BENZO(A)PYRENE	NT	●	●
BENZO(B)FLUORANTHENE	NT	●	●
BENZO(K)FLUORANTHENE	NT	●	*
DIBENZO(A,H)ANTHRACENE	NT	●	*
INDENO(1,2,3-CD)PYRENE	NT	●	●
NAPHTHALENE[5]	*	●	●
VOCs			
1,1,1,2-TETRACHLOROETHANE	*	NT	NT
1,1,2-TRICHLOROTRIFLUOROETHANE	NT	*	*
1,1-DICHLOROETHANE	●	*	*
1,1-DICHLOROETHENE	●	*	*
1,1-DICHLOROPROPENE	*	NT	NT

TABLE 10-1

ANALYTES WITH A DETECTED CONCENTRATION ABOVE THE REMEDIAL INVESTIGATION PALS[(1)] POTENTIAL SOURCE OF CONTAMINATION 45 NAS JACKSONVILLE JACKSONVILLE, FLORIDA

PARAMETER	Groundwater	Subsurface Soil	Surface Soil
1,2,3-TRICHLOROBENZENE	*	NT	NT
1,2,3-TRICHLOROPROPANE	*	NT	NT
1,2,4-TRIMETHYLBENZENE	*	NT	NT
1,2-DICHLOROETHANE	●	*	*
1,3,5*TRIMETHYLBENZENE	*	NT	NT
1,3-DICHLOROPROPANE	*	NT	NT
2,2-DICHLOROPROPANE	*	NT	NT
2-CHLOROTOLUENE	*	NT	NT
4-CHLOROTOLUENE	*	NT	NT
4-ISOPROPYLTOLUENE	*	NT	NT
2-BUTANONE	NT	*	*
2-HEXANONE	NT	*	*
4-METHYL-2-PENTANONE	NT	*	*
ACETONE	NT	*	*
BROMOBENZENE	*	NT	NT
BROMOCHLOROMETHANE	*	NT	NT
CARBON DISULFIDE	NT	*	*
CIS-1,2-DICHLOROETHENE	●	*	*
CYCLOHEXANE	NT	*	*
DIBROMOMETHANE	*	NT	NT
M+P-XYLENES	*	NT	NT
METHYL ACETATE	NT	*	*
METHYL CYCLOHEXANE	NT	*	*

TABLE 10-1

ANALYTES WITH A DETECTED CONCENTRATION ABOVE THE REMEDIAL INVESTIGATION PALS(1) POTENTIAL SOURCE OF CONTAMINATION 45 NAS JACKSONVILLE JACKSONVILLE, FLORIDA

PARAMETER	Groundwater	Subsurface Soil	Surface Soil
N-BUTYLBENZENE	*	NT	NT
N-PROPYLBENZENE	*	NT	NT
O-XYLENE	*	NT	NT
SEC-BUTYLBENZENE	*	NT	NT
TERT-BUTYLBENZENE	*	NT	NT
TETRACHLOROETHENE	●	●	●
TOTAL XYLENES	NT	*	*
TRICHLOROETHENE	●	*	*
TPH (C08-C40)(6)	NT	*	*

Notes:

(1) The soil appropriate RI PALs for the metals are the NAS Jacksonville background values (see Appendix B), if the NAS Jacksonville background values are greater than the FDEP or USEPA soil criteria. The appropriate FDEP criteria are identified in Chapter 62-777, Florida Administrative Code (F.A.C.), as the Groundwater Cleanup Target Levels (GCTLs) for groundwater; and SCTLs for soil. The appropriate USEPA criteria are the USEPA Regions 3, 6, and 9 Residential Regional Screening Levels (R-RSLs) for soil and the USEPA Regions 3, 6, and 9 RSLs for Tapwater (USEPA-TAP) for groundwater.

(2) "NT" indicates the analyte was not tested.

(3) "*" indicates that the parameter was analyzed for, but not detected above the appropriate PAL.

(4) Carcinogenic PAHs have a common toxicity mechanism, but display difference toxic potencies. The Toxic Equivalency Factors (TEF) approach is used to convert individual carcinogenic PAH site concentrations into a single concentration of the index chemical, benzo(a)pyrene (BAP) (FDEP, 2005; FDEP, 2006). This index is called the total benzo(a)pyrene equivalent, or BAP equivalent.

(5) Naphthalene in the groundwater column, identified under the SVOCs fraction, was reported as a VOC in the mobile laboratory reports.

(6) The TRPH was reported by the laboratory as TPH (C08-C40). The "TPH" indicates "total petroleum hydrocarbons" and the C08-C40 indicates the analysis reports the summary concentrations on hydrocarbons in the sample that had between 8 carbon atoms and 40 carbon atoms.

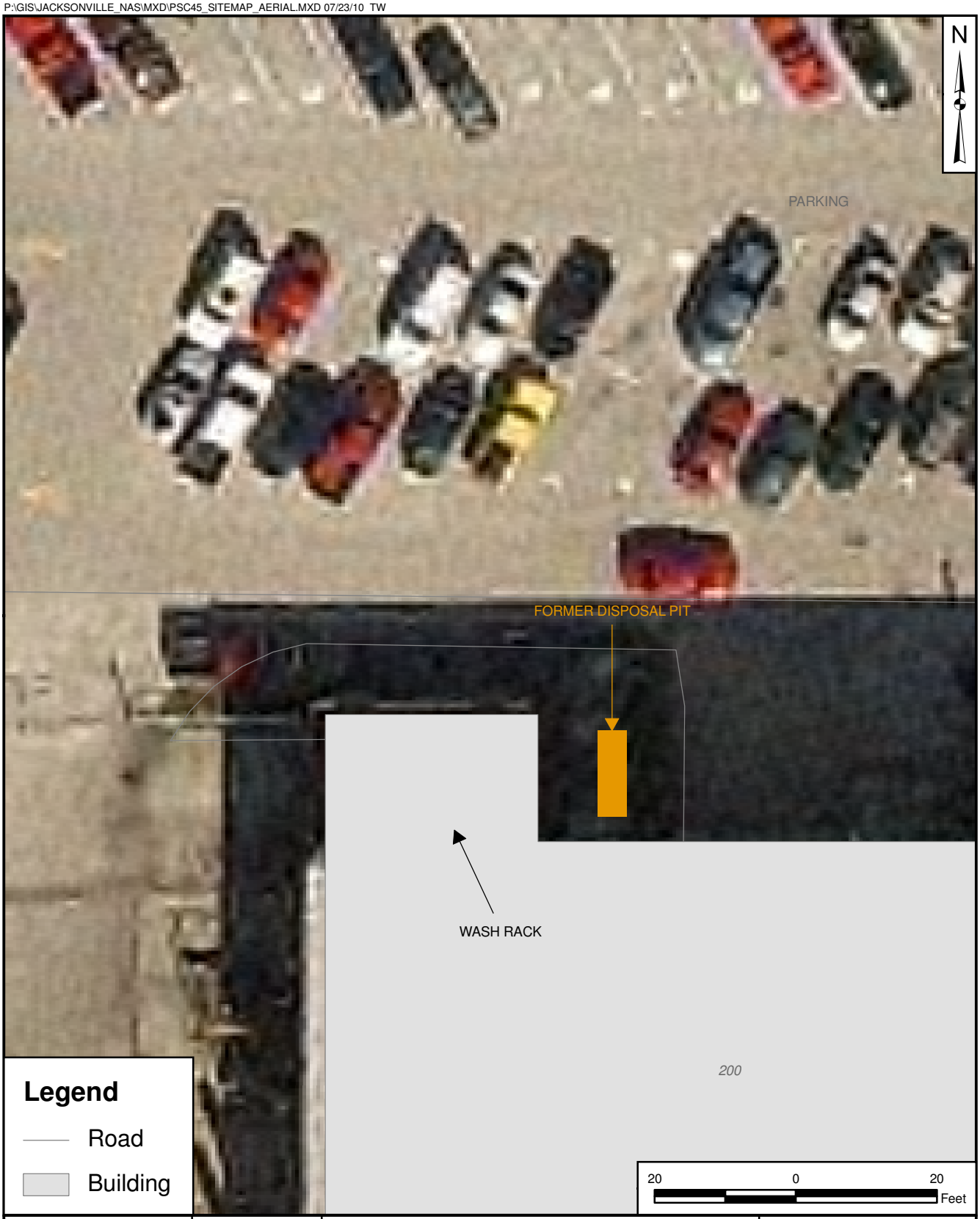
(7) "●" indicates analytes with a detected concentration above the RI PALs.

(8) Actual values detected are provided in Appendix A on Figures A-1 and A-2.

P:\GIS\JACKSONVILLE_NAS\MXD\JAX_VICINITY_MAP.MXD 07/23/10 TW

DRAWN BY T. WHEATON	DATE 07/23/10	NAVFAC	GENERAL LOCATION MAP NAS JACKSONVILLE JACKSONVILLE, FLORIDA	CONTRACT NUMBER CTO JM19
CHECKED BY B. PEEBLES	DATE 07/23/10			OWNER NUMBER —
REVISED BY —	DATE —			APPROVED BY — DATE —
SCALE AS NOTED				FIGURE NO. FIGURE 10-1 REV 0

P:\GIS\JACKSONVILLE_NAS\MXD\PSC45_SITEMAP_AERIAL.MXD 07/23/10 TW



DRAWN BY: S. PAXTON	DATE: 10/06/08	SITE MAP PSC 45 - WASH RACK AND FORMER DISPOSAL PIT NAS JACKSONVILLE JACKSONVILLE, FLORIDA	CONTRACT NUMBER: CTO JM19
CHECKED BY: B. PEEBLES	DATE: 07/23/10		APPROVED BY: ___ DATE: ___
REVISED BY: ___	DATE: ___		APPROVED BY: ___ DATE: ___
SCALE: AS NOTED			FIGURE NO.: FIGURE 10-2 REV: 0

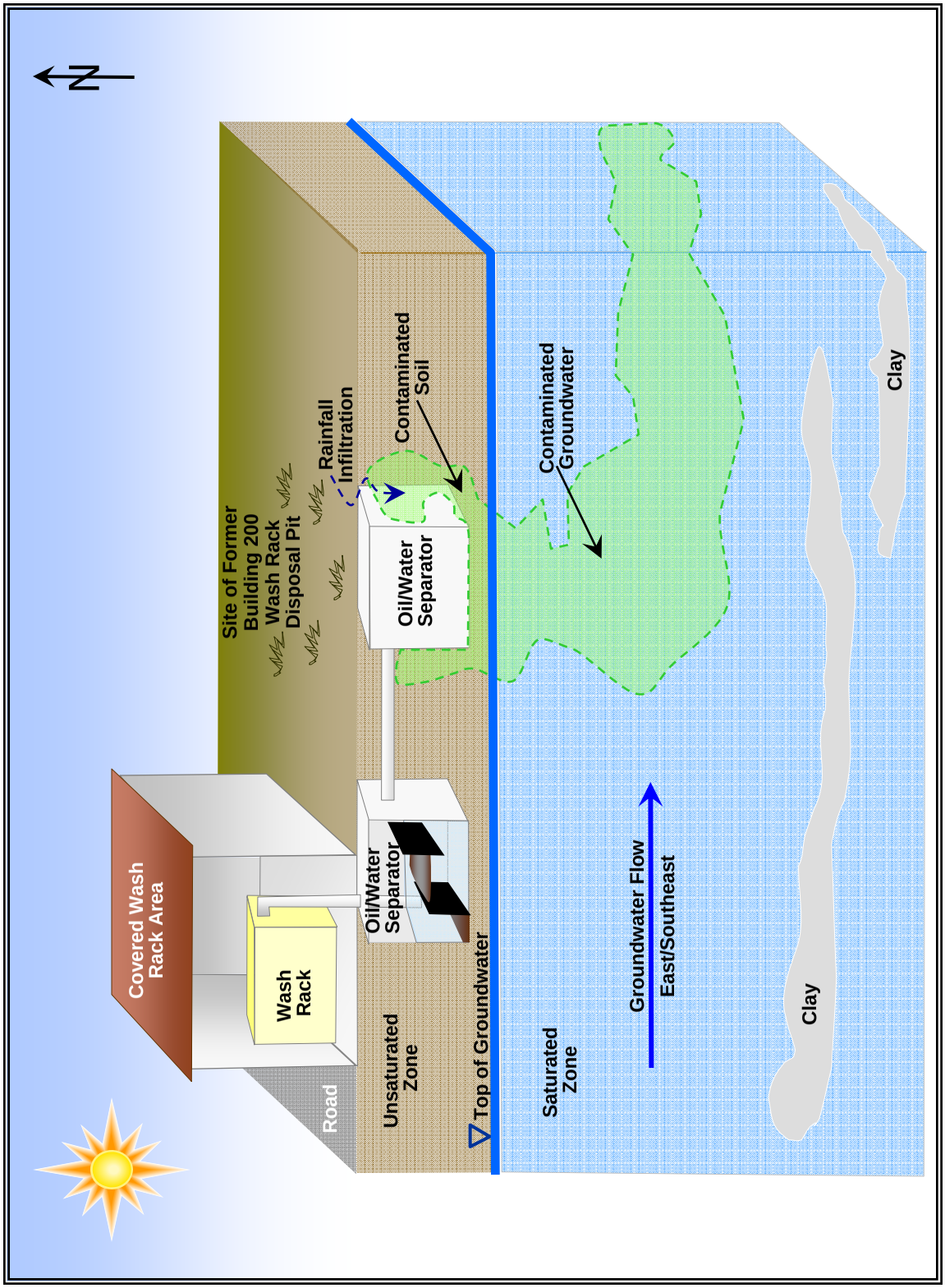
P:\GIS\JACKSONVILLE_NAS\MXD\BUILDING200_LAYOUT.MXD 07/27/10 TW

DRAWN BY	DATE
T.WHEATON	07/08/10
CHECKED BY	DATE
B. PEEBLES	07/27/10
COST/SCHED AREA	
SCALE	
AS NOTED	

NAVFAC

SITE LOCATION MAP
BUILDING 200
NAS JACKSONVILLE
JACKSONVILLE, FLORIDA

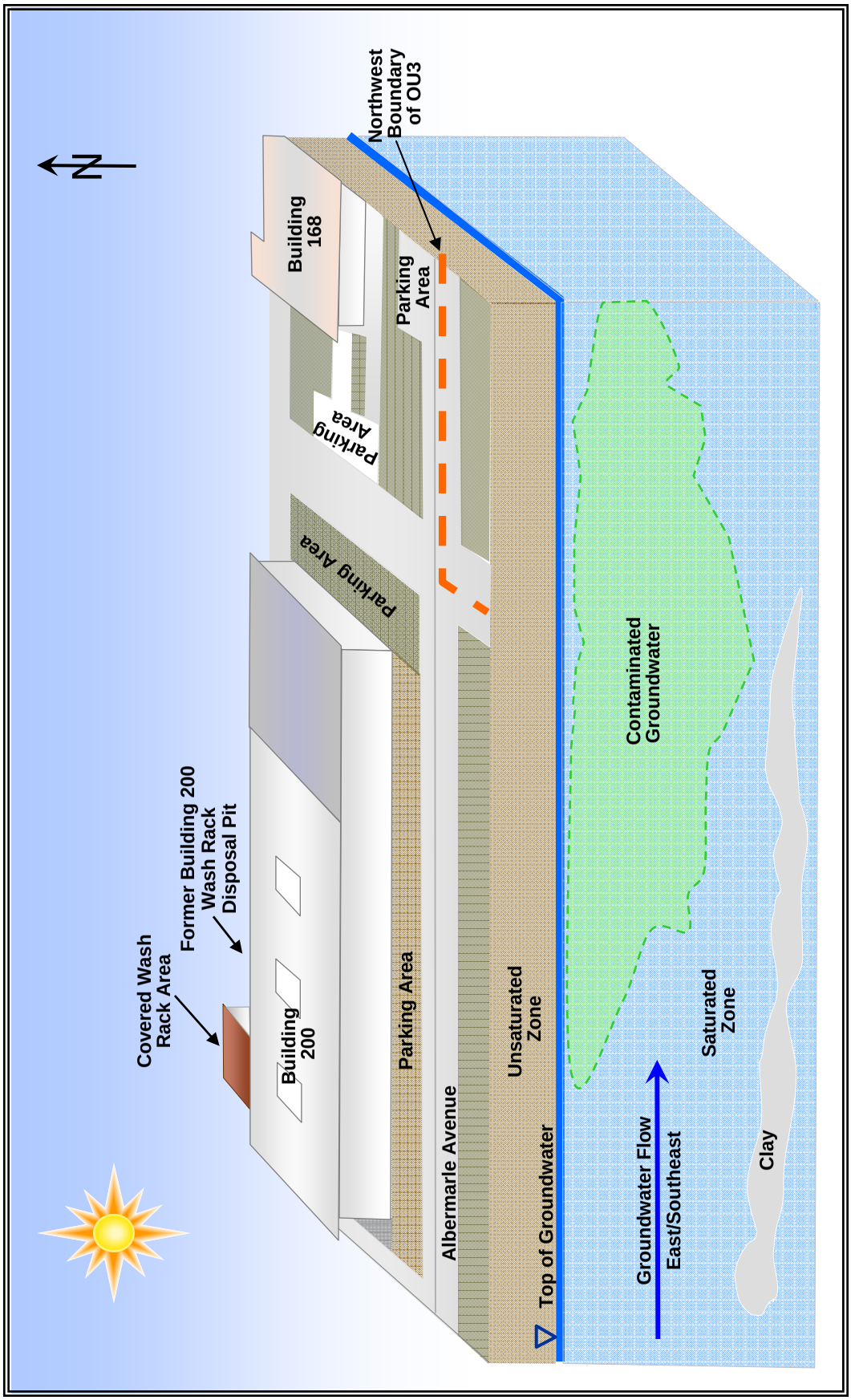
CONTRACT NUMBER	
CTO JM 19	
APPROVED BY	DATE
____	____
APPROVED BY	DATE
____	____
FIGURE NO.	REV
FIGURE 10-3	0



DRAWN BY C. PENNINGTON DATE
CHECKED BY B. PEEBLES DATE
REVISED BY P. CHURCHILL DATE
SCALE NOT TO SCALE

CONCEPTUAL SITE MODEL
PSC 45 BLDG 200 WASH RACK AREA
NAS JACKSONVILLE
JACKSONVILLE, FLORIDA

CONTRACT NO. JM 19
OWNER NO.
APPROVED BY DATE
DRAWING NO. FIGURE 10-4 REV.



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CHECKED BY B. PEEBLES DATE 8/23/2010
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CONCEPTUAL SITE MODEL
PSC 45 BLDG 200 GENERAL AREA
NAS JACKSONVILLE
JACKSONVILLE, FLORIDA

CONTRACT NO. CTO JM19
OWNER NO.
APPROVED BY DATE
DRAWING NO. FIGURE 10-5
REV.

SAP Worksheet #11 – Project Quality Objectives/Systematic Planning Process Statements
(UFP-QAPP Manual Section 2.6.1)

11.1 PROBLEM DEFINITION

During the SA, site-related contaminants were identified in environmental media at PSC 45 at concentrations that exceed the PALs developed for the SA. The nature and extent of contamination at PSC 45 was not determined during the SA. It is also unknown if site-related contaminants persist at levels that could pose an unacceptable level of human health risk. Therefore, an RI must be conducted at PSC 45. The purpose of the RI is to develop data that enables the Partnering Team to (a) determine the nature and extent of contamination at PSC 45, (b) evaluate human health risks through a human health risk assessment (HHRA), and (c) determine the follow-up activities that may be required in subsequent remedial activities. Upon receipt of the RI data, the Partnering Team will make decisions regarding the next steps, if any, which will be necessary to support the FS process.

11.2 INFORMATION INPUTS

To resolve the problem described in Section 11.1, the inputs presented below are needed.

1. Chemical Data: Soil and groundwater chemical data must be collected and analyzed to determine if target analytes are present in site media at concentrations greater than risk-based screening criteria, which are identified as the PSLs. The target analytes are identified in Worksheet #15. The sampling methods that must be utilized are identified in Worksheet #18, and the analytical methods are presented in Worksheet #19.

2. Field Parameters: Field investigation parameters for groundwater include dissolved oxygen, oxidation-reduction potential (ORP), pH, conductivity, temperature, and turbidity. These data must be collected in the field. The relevant Standard Operating Procedures (SOPs) are presented in Worksheet #21.

3. Groundwater Level Measurements: Synoptic groundwater levels must be measured in each monitoring well to determine the groundwater flow direction. These measurements must occur prior to sampling or 24 hours after sampling to avoid perturbation of the groundwater levels caused by sampling.

4. Sampling location coordinates must be measured in the field to sub-meter accuracy using a global positioning system (GPS) so the spatial boundaries of contamination can be accurately delineated.

The exposure point concentrations (EPCs) will be calculated for soil and groundwater using the methods described in Section 11.5, if data gaps do not prevent the development of the HHRA.

Project Screening Levels

Chemical data will be compared against current USEPA and FDEP residential subsurface soil and groundwater risk-based screening criteria to determine if there are potentially unacceptable levels of target analytes present in these environmental media. The PSLs are derived from the most conservative (lowest) of the following criteria for each media of concern, which are identified for each target analyte in Worksheet #15:

Soil

The soil PSLs are derived from the following:

- Florida Soil Cleanup Target Levels (SCTLs) per Chapter 62-777, F.A.C., Table 2 (Soil), direct exposure residential.
- JAX SS BKS (NAS Jacksonville surface soil background value); JAX SB BKG (NAS Jacksonville subsurface soil background value) (Appendix B; ABB-ES, 1996).
- USEPA Regions 3, 6, and 9 Regional Screening Levels (RSLs) for Chemical Contaminants at Superfund Sites – Residential (R-RSL) (USEPA, 2010).

Groundwater

The groundwater PSLs are derived from the following:

- Florida Groundwater Cleanup Target Levels (GCTLs) per Chapter 62-777, F.A.C., Table 1 (Groundwater).
- Florida Primary Drinking Water Standards per Chapter 62-550.310, F.A.C.
- Florida Secondary Drinking Water Standards per Chapter 62-550.320, F.A.C.
- USEPA Regions 3, 6, and 9 RSLs for Chemical Contaminants at Superfund Sites – Tap Water Values (USEPA, 2010).

11.3 STUDY BOUNDARIES

Groundwater, surface soil, and subsurface soil that have become, or have the potential to become, contaminated as a result of site-related releases of contaminants are the three populations of primary interest. Also of interest in this investigation are the soil and groundwater populations that are not contaminated, which bound the extent of contaminated media. For groundwater, the population of

primary interest is the aquifer that extends from the water table surface to 64 feet bgs (the vertical extent of contamination does not extend below 64 feet bgs). For soils, the population of primary interest is the unsaturated zone to the water table at approximately 6 feet in depth. The space occupied by these two populations present the physical dimensions of the soil exposure unit(s) (EU) and the groundwater EU(s) to which a receptor may be exposed to target analytes.

The horizontal boundary for surface and subsurface soil, with target analyte concentrations in excess of PSLs, is expected to be contained within an area bordered by the Wash Rack enclosure to the west, the northern edge of Building 200 to the south, a hypothetical north-to-south edge located 10 feet to the east, and the southern edge of the parking lot to the north (see Figure 10-2). The horizontal boundary for groundwater, with target analyte concentrations in excess of PSLs, is expected to be contained within an area bordered by the road running north and south of the western edge of Building 200, the southern side of Albemarle (which is directly south of Building 200), a hypothetical north-to-south edge located 200 feet to the east of Building 200 and the northern edge of the parking lot directly north of Building 200 (see Figure 10-3).

The vertical boundary for the subsurface soil population is the soil just above the top of the water table (water table plus 2 feet). These soils are not in the saturated zone and represent the bottom edge of the soil that a utility worker would come in contact with during a dry excavation. The vertical boundary for the groundwater population is 64 feet bgs and is based upon the results of the SA.

Temporal Boundaries: Conditions at PSC 45 are expected to remain stable for the duration of this project. Beyond that time frame, chemical concentrations in subsurface soil and groundwater may decrease because of dispersion, advection, degradation, or other physical, chemical, or biochemical attenuation mechanisms. Land use controls (LUCs) may be developed based upon the findings of the HHRA relative to the current land use. These findings will not be representative if the current land use patterns change.

11.4 ANALYTIC APPROACH

The analytical approach will include a phased evaluation to verify the results of the SA. The SA confirmed the migration of VOC constituents from the source area soils to groundwater; however, it did not determine if other SVOCs, metals, PCBs, and TRPH detected in source area soils have also impacted groundwater. It is presumed that due to the lower mobility of some of these constituents that any impacts to groundwater may be restricted in both concentration and extent. As a result, the first phase of evaluation will be to test groundwater in the vicinity of the source area for the full array of potential COCs via the installation and sampling of monitoring wells. During this phase, the extent of impacts to soil shall also be determined.

The second phase of investigation will be to complete the delineation of groundwater COCs identified in the first phase followed by verification through the installation and sampling of monitoring wells.

Separate decision rules have been created for estimating the nature and extent of contamination and human health risk. The potential risk to ecological receptors is fully addressed under the current scope of investigation at OU 3 (Tetra Tech, 2010) and the similarity of conditions between PSC 45 and OU 3 (both hydrological and contaminant type) and proximity of PSC 45 to OU 3 preclude the need to evaluate risk to ecological receptors solely from exposure to PSC 45 contaminants that may discharge into the St. Johns River via the storm sewer. If data gaps do not prevent the development of an HHRA, then current or future human health risk for the defined EUs will be calculated (see Decision Rule #3).

For the purpose of this RI SAP, the extent of contamination in groundwater and soil is determined by comparing the detected concentration of a target analyte to the PSL for that target analyte in the environmental medium in which it was detected. The extent of contamination is defined as the outer boundary circumscribing concentrations of all target analytes in a particular medium that exceed PSLs. This outer boundary for each target analyte is a line connecting the midpoints between sample locations that contain that target analyte detected at a concentration equal to or less than the PSL and sample locations that contain that target analyte detected at a concentration greater than the PSL.

The EPCs used in the HHRA (see Decision Rule #3) are developed differently for soil and groundwater. The current route of exposure, which is related to the types of activities that occur at an industrial site, is limited to dermal (direct) contact with soil, incidental ingestion, and inhalation by construction, maintenance or utility workers during excavation projects. Should a change in land use occur, then other human receptors may have the potential for exposure to contaminants. If land were transferred for unrestricted use, receptors could include future adult and child residents and recreationists. If sufficient soil quality data are available, the soil EPCs will be the 95 percent upper confidence limit (UCL) of the mean. The 95% UCL will be calculated using the most current version of ProUCL (http://www.epa.gov/esd/tsc/software.htm). The maximum concentration in the soil samples will become the EPC if sufficient soil quality data are not available. The EPC for groundwater will be the 95% UCL which will be calculated, using the most current version of ProUCL, from the groundwater data associated with the downgradient wells (see Figure 17-2). The direction of groundwater flow will be based upon a figure showing the potentiometric surface of the surficial aquifer. This figure will be developed after the wells shown on Figure 17-2 are installed.

Decision Rule #1
If the boundary of contamination (as described above) can be developed by the Partnering Team, then discontinue investigation of the extent of contamination. If this condition is not met, then the Partnering

Team will evaluate the data and develop an approach, which may include conducting additional sampling to refine the estimate of extent of contamination for PSC 45.

Decision Rule # 2

If data gaps do not prevent the development of an HHRA, then current and future human health risk for the defined EUs will be calculated (see Decision Rule #3). If this condition is not met, then the Navy will evaluate the data and develop an approach, which may include conducting additional sampling, to fill the data gaps.

Decision Rule # 3

If current or future human health risk for the defined EUs does not exceed the cumulative lifetime excess cancer risk level of $1.0E^{-6}$ for carcinogens or the hazard index of 1.0 for non-carcinogenic contaminants with the same target organ(s) systems, then recommend NFA. If this condition is not met, then the Navy will conduct an FS to evaluate remedial alternatives and an appropriate path forward for the site.

11.5 PERFORMANCE CRITERIA

Selection of sample locations were based on the results from the SA conducted by Tetra Tech in August 2009 are shown on Figures 17-1 and 17-2, in Worksheet #17. These sample locations were strategically placed to establish the volumes over which target analyte concentrations exceed the nature and extent PSLs and to obtain comprehensive spatial representation of the exposure units so that risks are unbiased or are possibly biased toward exaggerated risk, thus minimizing the chance of unknowingly leaving an unacceptable environmental condition without mitigation. The Partnering Team will use the results of the investigation based on this biased sampling approach to determine if the amount, type, and quality of data collected are sufficient to support the attainment of project objectives. This will include an evaluation to ensure that targeted method detection limits (MDLs), Limits of Detection (LODs), and limits of quantitation (LOQs) meet the quality specifications in Worksheet #15, and other data quality indicators (DQIs) meet the specifications of Worksheets #34 through #37. If all planned locations yield the intended data and the data are not compromised by quality deficiencies, then the data will be concluded to be suitable and sufficient for decision making.

11.6 PLAN FOR OBTAINING DATA

The sampling design and rationale for the PSC 45 RI are presented in Worksheet #17.

SAP Worksheet #12 – Measurement Performance Criteria Table Field Quality Control Samples
(UFP-QAPP Manual Section 2.6.2)

QC Sample	Analytical Group	Frequency	Data Quality Indicators (DQIs)	Measurement Performance Criteria (MPCs)	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
Equipment Rinsate Blanks	All Analytical Groups	One per 20 field samples per matrix per sampling equipment[1].	Bias/ Contamination	No analytes ≥ ½ LOQ, except common laboratory contaminants, which must be < LOQ.	S&A
Trip Blanks	VOCs	One per cooler containing VOC samples.	Bias/ Contamination	No analytes ≥ ½ LOQ, except common laboratory contaminants, which must be < LOQ.	S&A
Field Duplicate	All Analytical Groups	One per 10 field samples collected.	Precision	Values > 5X LOQ: Relative Percent Difference (RPD) must be ≤ 30% (aqueous) and ≤ 50% (solids)[2, 3].	S&A
Cooler Temperature Indicator	All Analytical Groups	One per cooler.	Representativeness	Temperature must be between 0 and 6 degrees Celsius (°C).	S

Notes:

1 Equipment rinsate blanks will be collected if non-dedicated equipment is used to collect samples. For disposable equipment, one sample per batch of disposable equipment will be collected.

2 If duplicate values for non-metals are < 5x LOQ, the absolute difference should be < 2x LOQ.

3 If duplicate values for metals are < 5x LOQ, the absolute difference should be < 4x LOQ.

SAP Worksheet #13 – Secondary Data Criteria and Limitations Table
(UFP-QAPP Manual Section 2.7)

Secondary Data	Data Source	Data Generator(s)	How Data Will Be Used	Limitations on Data Use
NAS Jacksonville Background Data PALs for Metals	RI/FS for OU 1 NAS Jacksonville	Originating Organization: ABB Environmental Services. March, 1996	Background data will be used to determine the PSL for metals, if the background concentration for that metal is above a listed state or federal PSL (see footnote # 1 in Table 10-1, notes to the tables in Worksheet #15, and Appendix B).	None.
Historical data on target analyte concentrations in soil and groundwater	SA event of August 2009 (results summarized in Appendix A)	Originating Organization: Tetra Tech Data Types: Soil and Groundwater Data Collection Dates: August 2009	These data were used to determine the locations of the RI samples (see Figures 17-1 and 17-2).	None.

SAP Worksheet #14 – Summary of Project Tasks
(UFP-QAPP Manual Section 2.8.1)

14.1 FIELD INVESTIGATION TASKS

The field activities include:

- Mobilization/Demobilization
- Health and Safety Training
- Utility Clearance/Dig Permits
- Monitoring Equipment Calibration
- Monitoring Well Installation
- Groundwater Sampling
- Soil Sampling
- Global Positioning System Locating and Surveying
- Investigation-Derived Waste (IDW) Management/Inspections
- Field Decontamination Procedure
- Field Documentation Procedures

Additional project activities include the following tasks:

- Documentation and Records
- Data Packages
- Data Review and Third Party Data Validation
- Analytical Tasks
- Data Management Tasks
- Assessment and Oversight
- Data Review
- Project Reports

14.2 MOBILIZATION/DEMOBILIZATION

Multiple mobilizations may be required to meet the project objectives. The mobilization shall consist of the delivery of all equipment, materials, and supplies to the site; the complete assembly in satisfactory working order of all such equipment at the site; and the satisfactory storage at the site of all such materials and supplies. Tetra Tech will coordinate with the Base to identify locations for the storage of equipment and supplies.

The demobilization shall consist of the prompt and timely removal of all equipment, materials, and supplies from the site following completion of the work. Demobilization includes the cleanup and removal of waste generated during the conduction of the investigation.

14.3 HEALTH AND SAFETY TRAINING

Site-specific Health and Safety Training to all Tetra Tech field staff and subcontractors will be provided as part of the site mobilization.

14.4 UTILITY CLEARANCE/DIG PERMITS

Prior to the commencement of any intrusive activities, Tetra Tech will coordinate utility clearance with the Base and Sunshine State One Call. The air station and utility companies subscribed to Sunshine State One Call will identify and mark-out utilities that may be present within the soil boring locations. The Tetra Tech FOL will also obtain a dig permit from the Public Works Department (PWD) at NAS Jacksonville.

14.5 MONITORING EQUIPMENT CALIBRATION

These procedures are described in Worksheet #22.

14.6 SOIL SAMPLING

During the initial mobilization, 10 soil borings at the Building 200 Wash Rack site will be advanced utilizing DPT techniques (Figure 17-1) for the purpose of delineating the nature and extent of impacts to shallow soil. Soil samples will be collected from the surface and subsurface soil which is from 6 inches to 2 feet above the top of the water table. This generates a total of 10 soil samples that will be collected for totals analyses. It should be noted, that additional step out samples may be required to fully delineate the extent of impact to shallow soils. Should additional samples be necessary, step out samples will be collected in order to establish a clean boundary at or below residential risk-based PALs. Soil sample step out locations will be field determined but in general will be the closest location available.

All of the soil samples will be collected using the procedures specified in FDEP SOP FS 3000 or Tetra Tech SOP SA-2.5 [Direct Push Technology (Geoprobe®/Hydropunch™)]. Worksheets #17 and #18 specify the soil sampling locations and analyte groups for this investigation. Worksheet #19 specifies the analytical methods to be used.

Additional step out borings will be conducted, if required, should results exceed RI PALs, utilizing the same sample intervals needed to complete soil delineations.

14.7 MONITORING WELL INSTALLATION AND SAMPLING

Well Installation

Permanent monitoring wells will be installed using DPT techniques, unless physical limitations of the area require a different technology. Shallow monitoring wells will be constructed of a 2-inch inside diameter, Schedule 40 flush-joint polyvinyl chloride (PVC) riser and flush-joint 0.010-inch factory-slotted well screen. Shallow monitoring well screen sections will be approximately 10 feet in length and positioned to intersect the water table. If required, the vertical extent monitoring well will be constructed of a 0.75-inch inside diameter, Schedule 40 flush-joint PVC riser and with approximately 5 feet of 0.010-inch, factory-slotted well screen. All well screens will be prepacked. After the DPT borings are pushed to the desired depth, wells will be installed through the DPT rods. Wells will be gauged to ensure they are installed to the proper depth. A monitoring well log will be prepared for each well location (see Appendix C).

Well Development and Sampling

The monitoring wells will be developed to remove fine sediment from around the screened interval of the well. Wells will be developed by pumping using either a submersible pump or peristaltic pump. Field parameters (pH, temperature, turbidity, and specific conductance) will be measured at equally-spaced time intervals during well development. Wells will be developed a maximum of one hour or until the field measurements become stable and the development water is visibly clear. Water quality stabilization will be determined using the following criteria:

- Temperature $\pm$ 0.2 degrees ºC
- pH $\pm$ 0.2 standard units
- Specific conductivity $\pm$ 5 percent of reading

The data will be recorded on a monitoring well development record (see Appendix C). No sooner than 24 hours after development, groundwater samples will be collected from monitoring wells in general accordance with to FDEP SOP 001/01 FS2200: Groundwater Sampling and FS1000. Prior to obtaining samples, synoptic water levels and total well depths will be measured and recorded on a groundwater level measurement sheet (see Appendix C). A second round of water levels will be collected no sooner than one month later and submitted on a separate field data sheet.

The wells will be purged using a peristaltic pump using low flow quiescent purging techniques per FDEP SOPs. The data will be recorded on a low flow purge data sheet (see Appendix C). Depending on the groundwater parameters, up to five well volumes may be purged. If wells are purged dry with less than one well volume removed, the water level in the well will be allowed to recover enough to collect three

field readings (pH, temperature, turbidity, dissolved oxygen, and specific conductance) prior to collecting a water sample. If the well does not purge dry using the low flow purging technique, groundwater characteristics will be taken (per FDEP SOP FS 2210) after each well volume of water is purged, or at 2- to 10-minute intervals, depending on the flow rate. Stabilization will be defined according to the following scenarios:

1) When purging a well that has a partially submerged well screen, a minimum of one well volume will be purged prior to collecting measurements of field parameters listed below. If the well screen is fully submerged, then a minimum of one volume of the pump, associated tubing, and flow cell will be purged prior to collecting field parameters listed below. Purging will be considered complete when three consecutive measurements in which the field parameters are within the desired limits as shown below.

   - Temperature ± 0.2° C
   - pH ± 0.2 Standard Units
   - Specific Conductivity ± 5 percent of reading
   - Dissolved oxygen is not greater than 20 percent of saturation at the field measured temperature
   - Turbidity is not greater than 20 Nephelometric Turbidity Units (NTUs)

2) When purging a well and the stabilization described in 1) above cannot be achieved, three consecutive measurements of the following parameters are required:

   - Dissolved oxygen ± 0.2 mg/L or 10 percent, whichever is greater
   - Temperature ± 0.2° C
   - pH ± 0.2 Standard Units.
   - Specific Conductivity ± 5 percent of reading
   - Turbidity ± 5 NTUs or 10 percent, whichever is greater

If stabilization is not achieved, five screen volumes must be removed prior to samples being collected in the appropriate sample containers. Samples to be analyzed for volatile constituents will be collected first and immediately sealed in 40-milliliter (mL) vials so that no headspace exists. The data acquired during sampling will be recorded on a groundwater sample log sheet (see Appendix C).

Since data obtained from groundwater during the SA did not include all constituents detected in soils, a shallow groundwater monitoring well will be installed at the immediate downgradient edge of the former disposal pit for the purpose of determining if groundwater is impacted as a result of contamination bound up in shallow soils. The monitoring well will be a 2-inch inside diameter, Schedule 40 flush-joint PVC riser and flush-joint 0.010-inch factory-slotted well screen, installed using DPT techniques. The

monitoring well will be developed and sampled for the full range of constituents analyzed in soil including metal, PCB, TRPH, VOC, and SVOC constituents as shown in Worksheet #18.

14.8 DPT GROUNDWATER SAMPLING

During the initial mobilization, groundwater sampling will be conducted at the PSC 45 via DPT techniques. Seven groundwater sample locations are associated with PSC 45 (see Figure 17-2). Actual DPT sampling locations may need to be relocated based on physical obstructions or utilities. Field methodologies for DPT groundwater sample collection are detailed below.

Groundwater samples will be collected using a DPT groundwater sampling system in conjunction with a peristaltic pump and sterile Teflon® and medical-grade silicon tubing. In general, the DPT groundwater sampling system consists of an enclosed 4-foot groundwater sampler attached to 2.125-inch outside diameter steel drive rods, which are hammer driven via DPT to the maximum desired sampling depth (approximately 64 feet bgs).

When the desired sampling depth is reached, the outer sleeve of the groundwater sampler is retracted to expose a 4-foot mill-slotted (0.02-inch) well point screen to the formation. Teflon® tubing will then be lowered through the inner core of the DPT drive rod to the bottom of the borehole and attached to a peristaltic pump using silicon tubing. To minimize sediment loading, the tubing will be placed 2.5 feet from the bottom of each borehole in the center of the screen. A sample will be collected once the purge water becomes visibly clear. If the purge water does not become visibly clear due to fine sediments in this area, purging should be conducted in accordance with the methods in FDEP SOP FS 2200 (see Worksheet #21) before a sample is collected to reduce turbidity.

Groundwater samples, at each of the 7 locations, will be collected from 4 intervals, yielding a total of 28 field samples. The sample intervals are:

- 12 to16 feet bgs
- 20 to 24 feet bgs
- 40 to 44 feet bgs
- 60 to 64 feet bgs

The actual sampling depth at each boring location is subject to change based on the lithology boring data. If any significant clay units (greater than 2-feet thick) are found, then a groundwater sample will be collected from the interval immediately above it. The boring will not be advanced through a significant clay unit.

Due to the nature of the sample collection, geochemical parameters and turbidity will not be measured during this sampling event. DPT groundwater samples will be collected and submitted to Katahdin for analysis.

All sample locations will be marked with a wooden stake, brightly colored pin flag, or spray marking paint indicating the sample location. Coordinates will be determined by GPS at each individual sample location, which will allow for future repeatable investigations or guide in any remedial action. All removable sample location markers will be removed prior to the final demobilization.

All of the groundwater samples will be collected using the procedures specified in FDEP SOP FS 2200 for groundwater sampling (FDEP, 2008). Worksheets #17 and #18 specify the soil sampling locations and analyte groups for this investigation. Worksheet #19 specifies the analytical methods to be used. It should be noted that DPT sample locations will be tested for VOCs.

14.9 GLOBAL POSITION SYSTEM LOCATING

A GPS unit will be used to locate all sampling points in accordance with Tetra Tech SOP SOP-01 (Appendix C). The GPS equipment will be checked on control monuments before and after day's use, and these checks will be documented in the field notebook. To ensure sub-meter accuracy, a minimum of six satellites are required to capture a position.

14.10 INVESTIGATION-DERIVED WASTE MANAGEMENT/INSPECTIONS

Types of IDW generated during this investigation that could be potentially contaminated include: groundwater and excess soil collected but not placed in the laboratory supplied sample containers, sampling equipment, decontamination wastewaters, and personal protective equipment (PPE) and clothing. Groundwater displaced during this investigation and excess soil will be placed into labeled, sealable 55-gallon steel drums provided by NAS Jacksonville PWD. The drums will be inspected weekly until picked up and transported by the PWD to a secured area designated by the Navy. Proper disposal of these wastes will be performed by the Navy (or its designee) after the analytical results of the groundwater and soil samples are received from the laboratory and reviewed. PPE and clothing will be wiped clean and disposed of in trash containers as general refuse.

14.11 FIELD DECONTAMINATION PROCEDURE

Decontamination of major equipment and sampling equipment will be in general accordance with FDEP SOP FC 1000 (FDEP, 2008).

14.12 FIELD DOCUMENTATION PROCEDURES

Pre-preserved, certified-clean bottle ware will be supplied by Katahdin. Matrix-specific sample log sheets will be maintained for each sample collected. In addition, sample collection information will be recorded

in bound field notebooks or specific field forms. Samples will be packaged and shipped according to FDEP SOP FS 1000 (FDEP, 2008).

Field documentation will be performed in accordance with Tetra Tech SOP SA-6.3 (see Appendix C). A summary of all field activities will be properly recorded in indelible ink in a bound logbook with consecutively numbered pages that cannot be removed. Logbooks will be assigned to field personnel and will be stored in a secured area when not in use.

At a minimum, the following information will be recorded in the site logbook:

- Name of the person to whom the logbook is assigned.
- Project name.
- Project start date.
- Names and responsibilities of on-site project personnel including subcontractor personnel.
- Arrival/departure of site visitors.
- Arrival/departure of equipment.
- Sampling activities and sample log sheet references.
- Description of subcontractor activities.
- Sample pick-up information including chain-of-custody numbers, air bill numbers, carrier, time, and date.
- Description of borehole or monitoring well installation activities and operations.
- Health and safety issues.
- Description of photographs including date, time, photographer, roll and picture number, location, and compass direction of photograph.

All entries will be written in indelible ink and no erasures will be made. If an incorrect entry is made, striking a single line through the incorrect information will make the correction; the person making the correction will initial and date the change.

14.13 DOCUMENTATION AND RECORDS

Documentation of sample location coordinates, borings logs, chain-of-custody forms, samples logs, and shipping documents for all samples will be recorded and kept. Also, electronic and hardcopies of the final SAP will be kept on-site and in the Tetra Tech project files.

14.14 DATA PACKAGES

Data packages will include receipt of analytical data packages from Katahdin, and generation of Tetra Tech data validation reports.

14.15 DATA REVIEW TASKS AND THIRD PARTY DATA VALIDATION

Katahdin will verify that all samples listed on the chain-of-custody are analyzed in accordance with methods specified on the chain-of-custody form, the laboratory scope of work, and in this SAP. Data verification and validation will be performed by Tetra Tech as described in Worksheets #35 and #36. A data validation report will be produced for each Sample Delivery Group (SDG).

All field data records and validated data will be reviewed by Tetra Tech personnel to determine the usability of the data (see Worksheet #37). The outcome of this assessment will be conveyed to the Partnering Team for agreement before the project report is finalized. Data limitations pertaining to Project Quality Objectives (PQOs) and PSLs will be identified, and corrective actions will be taken as necessary.

14.16 ANALYTICAL TASKS

Chemical analysis will be performed off-site by Katahdin. Katahdin is a current Department of Defense (DoD) Environmental Laboratory Accreditation Program (ELAP) accredited laboratory. In addition, Katahdin holds National Environmental Laboratory Accreditation Program (NELAP) accreditation with the State of Florida Department of Health serving as their primary accrediting authority. Copies of the pertinent laboratory accreditation are located in Appendix D. Analyses will be performed in accordance with the analytical methods identified in Worksheet #19. Katahdin will meet the PSLs as shown in Worksheet #15. Katahdin will perform chemical analysis following laboratory specific SOPs (Worksheets #19 and #23) developed based on the analytical methods listed in Worksheet #19 and #30. Copies of the laboratory SOPs are included in Appendix D.

All soil results will be reported by the laboratory on a dry-weight basis. Results of percent moisture will be reported in each analytical data package and electronic data files. This information will also be captured in the project database which will eventually be uploaded to Naval Installation Restoration Information Solution (NIRIS). Percent moisture information will also be captured in the data report.

The analytical data packages provided by Katahdin will be in a Contract Laboratory Program (CLP)-like format and will be fully validatable and contain raw data, summary forms for all sample and laboratory method blank data, and summary forms containing all method specific quality control (QC) information [results, recoveries, RPD, relative standard deviations (RSDs), and/or percent difference drift (%D), etc.].

14.17 DATA MANAGEMENT TASKS

- Project documentation and records
  - Field sample collection and field measurement records are described in Worksheets #27 and #29.
  - Laboratory data package deliverables are described in the analytical specifications.
  - Data assessment documents and records are listed in Worksheet #29.
- Data recording formats are described in Worksheet #27.

Data Handling and Management – After the field investigation is completed, the field sampling log sheets will be organized by date and media and filed in the project files. The field logbooks for this project will be used only for these sites and will also be categorized and maintained in the project files after the completion of the field program. Project personnel completing concurrent field sampling activities may maintain multiple field logbooks. When possible, logbooks will be segregated by sampling activity. The field logbooks will be titled based on date and activity. The data handling procedures to be followed by the laboratories will meet the requirements of the technical specification. The electronic data results will be automatically downloaded into the Tetra Tech database in accordance with proprietary Tetra Tech processes.

Data Tracking and Control – The Tetra Tech PM (or designee) is responsible for the overall tracking and control of data generated for the project.

- Data Tracking: Data is tracked from its generation to its archiving in the Tetra Tech project-specific files. The Tetra Tech Project Chemist (or designee) is responsible for tracking the samples collected and shipped to the subcontracted laboratory. Upon receipt of the data packages from the analytical laboratory, the Tetra Tech Project Chemist will oversee the data validation effort, which includes verifying that the data packages are complete and results for all samples have been delivered by the analytical laboratory.

- Data Storage, Archiving, and Retrieval: The data packages received from the subcontracted laboratory are tracked in the data validation logbook. After the data are validated, the data packages are entered into the Tetra Tech CLEAN file system and archived in secure files. The field records including field logbooks, sample logs, chain-of-custody records, and field calibration logs will be submitted by the Tetra Tech FOL to be entered into the CLEAN file system prior to archiving in secure project files. The project files are audited for accuracy and completeness. At the completion of the Navy contract, the records will be stored by Tetra Tech and eventually handed over to NAVFAC SE.

- Data Security: The Tetra Tech project files are restricted to designated personnel only. Records can only be borrowed temporarily from the project file using a sign-out system. The Tetra Tech Data Manager maintains the electronic data files. Access to the data files is restricted to qualified personnel only. File and data backup procedures are routinely performed.

14.18 ASSESSMENT AND OVERSIGHT

Refer to Worksheet #32 for assessment findings and Worksheet #33 for QA management reports.

14.19 DATA REVIEW

Data verification is described in Worksheet #34. Data validation is described in Worksheets #35 and #36. Usability assessment is described in Worksheet #37.

14.20 PROJECT REPORTS

The RI report for PSC 45 will be prepared to document the results from the investigation and the risk assessments. The report will include appropriate sections concerning RI activities, physical characteristics, nature and extent of contamination, risk to receptors, conclusions, and recommendations.

The draft RI report will be issued in draft to the NAS Jacksonville Partnering team for review. All comments will be addressed and a response to comments document will be prepared and submitted to the NAS Jacksonville Partnering Team with the draft-final RI report.

SAP Worksheet #15 – Reference Limits and Evaluation Table

(UFP-QAPP Manual Section 2.8.1)

NOTE: PSLs that, if exceeded, indicate an unacceptable level of human health risk are cumulative lifetime excess cancer risk level = $1E^{-6}$ and Hazard Index = 1.0 (common target organ effect). PSLs which, if exceeded, indicate the presence of contamination and will be used to delineate contamination. These PSLs are presented in the tables below.

Matrix: Soil
Analytical Group: Metals

Analyte	CAS Number	PSL (mg/kg)	PSL Reference	Project Quantitation Limit Goal (mg/kg)	Katahdin		
					LOQ (mg/kg)	LOD (mg/kg)	MDL (mg/kg)
ALUMINUM	7429-90-5	1,340	JAX SS BKS	447	30	10	1.6
ANTIMONY	7440-36-0	0.66	USEPA RBRSL	0.22	0.8	0.5	0.11
ARSENIC	7440-38-2	0.0013	USEPA RBRSL	0.00043	0.8	0.5	0.11
BARIUM	7440-39-3	11.2	JAX SS BKS	3.7	0.5	0.4	0.026
BERYLLIUM	7440-41-7	0.49	JAX SB BKG	0.16	0.5	0.05	0.0085
CADMIUM	7440-43-9	1.4	USEPA RBRSL	0.47	1	0.3	0.0084
CALCIUM	7440-70-2	668.3	JAX SB BKG	223	10	8	1.1
CHROMIUM	7440-47-3	6.6	JAX SS BKS	2.2	1.5	0.4	0.032
COBALT	7440-48-4	0.49	USEPA RBRSL	0.16	3	0.2	0.019
COPPER	7440-50-8	5.8	JAX SS BKS	1.9	2.5	1	0.073
IRON	7439-89-6	640	USEPA RBRSL	213	10	8	0.3
LEAD	7439-92-1	6.46	JAX SB BKG	2.2	0.5	0.4	0.1
MAGNESIUM	7439-95-4	99.8	JAX SS BKS	33.3	10	8	0.36
MANGANESE	7439-96-5	6.9	JAX SB BKG	2.3	0.5	0.4	0.058
MERCURY	7439-97-6	0.03	USEPA RBRSL	0.01	0.04	0.005	0.0011
NICKEL	7440-02-0	11	JAX SS BKS	3.7	4	0.4	0.054
POTASSIUM	7440-09-7	450.67	JAX SB BKG	150	100	40	10
SELENIUM	7782-49-2	0.95	USEPA RBRSL	0.32	1	0.7	0.39
SILVER	7440-22-4	1.6	USEPA RBRSL	0.53	1.5	0.5	0.049
SODIUM	7440-23-5	288	JAX SS BKS	96	100	50	1.7
THALLIUM	7440-28-0	0.42	JAX SS BKS	0.14	1.5	0.5	0.16
VANADIUM	7440-62-2	3.8	JAX SS BKS	1.3	2.5	0.4	0.035
ZINC	7440-66-6	14.49	JAX SB BKG	4.8	2.5	1.2	0.063

Notes:

CAS – Chemical Abstracts Service
mg/kg – Milligrams per kilogram

Project Screening Level Reference: JAX SS BKS (NAS Jacksonville surface soil background value); JAX SB BKG (NAS Jacksonville subsurface soil background value) (ABB-ES, 1996); USEPA RBRSL (USEPA Regions 3, 6, and 9 Risk-based Regional Screening Level - Protection of Groundwater, Dilution Attenuation Factor (DAF)=1); USEPA RSL (USEPA Regions 3, 6, and 9 Residential Regional Screening Level).

Bolded compounds indicate that the PSL is between the laboratory LOQ and LOD. The Partnering Team has agreed to accept this data for decision making if results less than the LOQ are "J" qualified and their impact on the attainment of project objectives will be described in the uncertainties section of the Risk Assessment.

Shaded and Bold compounds indicate the PSL is less than the LOD. The Partnering Team has agreed to replace the PSLs with the laboratory LOQs for decision making purposes, as suggested in "Guidance for the Selection of Analytical Methods for the Evaluation of Practical Quantitation Limits" (FDEP, 2004).

Matrix: Soil
Analytical Group: PCBs

Analyte	CAS Number	PSL (mg/kg)	PSL Reference	Project Quantitation Limit Goal (mg/kg)	Katahdin		
					LOQ (mg/kg)	LOD (mg/kg)	MDL (mg/kg)
AROCLOR-1016	12674-11-2	0.092	USEPA RBRSL	0.031	0.0034	0.0017	0.0012
AROCLOR-1221	11104-28-2	0.00012	USEPA RBRSL	0.00004	0.0034	0.0017	0.0016
AROCLOR-1232	11141-16-5	0.00012	USEPA RBRSL	0.00004	0.0034	0.0017	0.0019
AROCLOR-1242	53469-21-9	0.0053	USEPA RBRSL	0.0018	0.0034	0.0017	0.0012
AROCLOR-1248	12672-29-6	0.0052	USEPA RBRSL	0.0017	0.0034	0.0017	0.0012
AROCLOR-1254	11097-69-1	0.0088	USEPA RBRSL	0.0029	0.0034	0.0017	0.0009
AROCLOR-1260	11096-82-5	0.024	USEPA RBRSL	0.008	0.0034	0.0017	0.0012
TOTAL AROCLOR	1336-36-3	0.22	USEPA RSL	0.073	1	1	1

1 Laboratory reports individual Aroclor values

Project Screening Level Reference: JAX SS BKS (NAS Jacksonville surface soil background value); JAX SB BKG (NAS Jacksonville subsurface soil background value) (ABB-ES, 1996); USEPA RBRSL (USEPA Regions 3, 6, and 9 Risk-based Residential Screening Level - Protection of Groundwater); USEPA RSL (USEPA Regions 3, 6, and 9 Residential Regional Screening Level).

Shaded and Bold compounds indicate the PSL is less than the LOD. The Partnering Team has agreed to replace the PSLs with the laboratory LOQs for decision making purposes, as suggested in "Guidance for the Selection of Analytical Methods for the Evaluation of Practical Quantitation Limits" (FDEP, 2004).

Matrix: Soil
Analytical Group: VOCs

Analyte	CAS Number	PSL (mg/kg)	PSL Reference	Project Quantitation Limit Goal (mg/kg)	Katahdin		
					LOQ (mg/kg)	LOD (mg/kg)	MDL (mg/kg)
1,1,1-TRICHLOROETHANE	71-55-6	3.2	USEPA RBRSL	1	0.005	0.0025	0.00042
1,1,2,2-TETRACHLOROETHANE	79-34-5	0.000026	USEPA RBRSL	0.000009	0.005	0.0025	0.00084
1,1,2-TRICHLOROETHANE	79-00-5	0.000078	USEPA RBRSL	0.00003	0.005	0.0025	0.00097
1,1,2-TRICHLOROTRIFLUOROETHANE	76-13-1	150	USEPA RBRSL	50	0.005	0.0025	0.0009
1,1-DICHLOROETHANE	75-34-3	0.00069	USEPA RBRSL	0.0002	0.005	0.0025	0.0017
1,1-DICHLOROETHENE	75-35-4	0.12	USEPA RBRSL	0.04	0.005	0.0025	0.00093
1,2,4-TRICHLOROBENZENE	120-82-1	0.0068	USEPA RBRSL	0.002	0.005	0.0025	0.00079
1,2-DIBROMO-3-CHLOROPROPANE	96-12-8	0.00000014	USEPA RBRSL	0.00000005	0.005	0.0025	0.0015
1,2-DIBROMOETHANE	106-93-4	0.0000018	USEPA RBRSL	0.0000006	0.005	0.0025	0.0012
1,2-DICHLOROBENZENE	95-50-1	0.36	USEPA RBRSL	0.1	0.005	0.0025	0.00078
1,2-DICHLOROETHANE	107-06-2	0.000042	USEPA RBRSL	0.00001	0.005	0.0025	0.001
1,2-DICHLOROPROPANE	78-87-5	0.00013	USEPA RBRSL	0.00004	0.005	0.0025	0.0014
1,3-DICHLOROBENZENE	541-73-1	7	FDEP LSCTL	130	0.005	0.0025	0.00062
1,4-DICHLOROBENZENE	106-46-7	0.00041	USEPA RBRSL	0.0001	0.005	0.0025	0.00044
2-BUTANONE	78-93-3	1.5	USEPA RBRSL	0.5	0.025	0.0125	0.0059
2-HEXANONE	591-78-6	0.011	USEPA RBRSL	0.004	0.025	0.0125	0.0048
4-METHYL-2-PENTANONE	108-10-1	0.45	USEPA RBRSL	0.2	0.025	0.0125	0.0059
ACETONE	67-64-1	4.5	USEPA RBRSL	2	0.025	0.0125	0.0051
BENZENE	71-43-2	0.00021	USEPA RBRSL	0.00007	0.005	0.0025	0.00092
BROMODICHLOROMETHANE	75-27-4	0.000032	USEPA RBRSL	0.00001	0.005	0.0025	0.0006
BROMOFORM	75-25-2	0.0023	USEPA RBRSL	0.0008	0.005	0.0025	0.0007
BROMOMETHANE	74-83-9	0.0022	USEPA RBRSL	0.0007	0.01	0.005	0.0011
CARBON DISULFIDE	75-15-0	0.31	USEPA RBRSL	0.1	0.005	0.0025	0.00078
CARBON TETRACHLORIDE	56-23-5	0.00017	USEPA RBRSL	0.00006	0.005	0.0025	0.0013
CHLOROBENZENE	108-90-7	0.062	USEPA RBRSL	0.02	0.005	0.0025	0.00051
CHLORODIBROMOMETHANE	124-48-1	0.000039	USEPA RBRSL	0.00001	0.005	0.0025	0.001
CHLOROETHANE	75-00-3	0.06	FDEP LSCTL	1	0.01	0.005	0.0013
CHLOROFORM	67-66-3	0.000053	USEPA RBRSL	0.00002	0.005	0.0025	0.00035
CHLOROMETHANE	74-87-3	0.049	USEPA RBRSL	0.02	0.01	0.005	0.0014
CIS-1,2-DICHLOROETHENE	156-59-2	0.11	USEPA RBRSL	0.04	0.005	0.0025	0.00091
CIS-1,3-DICHLOROPROPENE	10061-01-5	0.00015	USEPA RBRSL	0.00005	0.005	0.0025	0.00072
CYCLOHEXANE	110-82-7	13	USEPA RBRSL	4	0.005	0.0025	0.0014
DICHLORODIFLUOROMETHANE	75-71-8	0.61	USEPA RBRSL	0.2	0.01	0.005	0.00092

Analyte	CAS Number	PSL (mg/kg)	PSL Reference	Project Quantitation Limit Goal (mg/kg)	Katahdin		
					LOQ (mg/kg)	LOD (mg/kg)	MDL (mg/kg)
ETHYLBENZENE	100-41-4	0.0017	USEPA RBRSL	0.0006	0.005	0.0025	0.00065
ISOPROPYLBENZENE	98-82-8	1.1	USEPA RBRSL	0.4	0.005	0.0025	0.00092
METHYL ACETATE	79-20-9	7.5	USEPA RBRSL	3	0.005	0.0025	0.0027
METHYL CYCLOHEXANE	108-87-2	NC	None	NC	0.005	0.0025	0.00096
METHYL TERT-BUTYL ETHER	1634-04-4	0.0028	USEPA RBRSL	0.0009	0.005	0.0025	0.0011
METHYLENE CHLORIDE	75-09-2	0.0012	USEPA RBRSL	0.0004	0.025	0.0125	0.0079
STYRENE	100-42-5	1.8	USEPA RBRSL	0.6	0.005	0.0025	0.00051
TETRACHLOROETHENE	127-18-4	0.000049	USEPA RBRSL	0.00002	0.005	0.0025	0.0012
TOLUENE	108-88-3	1.6	USEPA RBRSL	0.5	0.005	0.0025	0.0014
TOTAL XYLENES	1330-20-7	0.2	USEPA RBRSL	0.07	0.015	0.0075	0.0013
TRANS-1,2-DICHLOROETHENE	156-60-5	0.031	USEPA RBRSL	0.01	0.005	0.0025	0.00071
TRANS-1,3-DICHLOROPROPENE	10061-02-6	1.7	USEPA RSL	0.6	0.005	0.0025	0.00086
TRICHLOROETHENE	79-01-6	0.00072	USEPA RBRSL	0.0002	0.005	0.0025	0.00059
TRICHLOROFLUOROMETHANE	75-69-4	0.83	USEPA RBRSL	0.3	0.01	0.005	0.00091
VINYL CHLORIDE	75-01-4	0.0000056	USEPA RBRSL	0.000002	0.01	0.005	0.0013

NC = No criteria

Project Screening Level Reference: JAX SS BKS (NAS Jacksonville surface soil background value); JAX SB BKG (NAS Jacksonville subsurface soil background value) (ABB-ES, 1996); USEPA RBRSL (USEPA Regions 3, 6, and 9 Risk-based Regional Screening Level - Protection of Groundwater); USEPA RSL (USEPA Regions 3, 6, and 9 Residential Regional Screening Level); FDEP LSCTL (FDEP Chapter 62-777, F.A.C Soil Cleanup Target Levels, Leachability based on Groundwater Criteria.

Bolded compounds indicate that the PSL is between the laboratory LOQ and LOD. The Partnering Team has agreed to accept this data for decision making if results less than the LOQ are "J" qualified and their impact on the attainment of project objectives will be described in the uncertainties section of the Risk Assessment.

Shaded and Bold compounds indicate the PSL is less than the LOD. The Partnering Team has agreed to replace the PSLs with the laboratory LOQs for decision making purposes, as suggested in "Guidance for the Selection of Analytical Methods for the Evaluation of Practical Quantitation Limits" (FDEP, 2004).

Matrix: Soil
Analytical Group: SVOCs (including Low Level PAHs by SIM [1])

Analyte	CAS Number	PSL (mg/kg)	PSL Reference	Project Quantitation Limit Goal (mg/kg)	Katahdin		
					LOQ (mg/kg)	LOD (mg/kg)	MDL (mg/kg)
1,1-BIPHENYL	92-52-4	19	USEPA RBRSL	6	0.33	0.25	0.073
2,4,5-TRICHLOROPHENOL	95-95-4	14	USEPA RBRSL	5	0.82	0.62	0.155
2,4,6-TRICHLOROPHENOL	88-06-2	0.023	USEPA RBRSL	0.008	0.33	0.25	0.155
2,4-DICHLOROPHENOL	120-83-2	0.13	USEPA RBRSL	0.04	0.33	0.25	0.15
2,4-DIMETHYLPHENOL	105-67-9	0.86	USEPA RBRSL	0.3	0.33	0.25	0.165
2,4-DINITROPHENOL	51-28-5	0.082	USEPA RBRSL	0.03	0.82	0.62	0.377
2,4-DINITROTOLUENE	121-14-2	0.00029	USEPA RBRSL	0.0001	0.33	0.25	0.085
2,6-DINITROTOLUENE	606-20-2	0.05	USEPA RBRSL	0.02	0.33	0.25	0.079
2-CHLORONAPHTHALENE	91-58-7	15	USEPA RBRSL	5	0.33	0.25	0.087
2-CHLOROPHENOL	95-57-8	0.15	USEPA RBRSL	0.05	0.33	0.25	0.164
2-METHYLNAPHTHALENE (1)	91-57-6	0.75	USEPA RBRSL	0.3	0.02	0.01	0.0022
2-METHYLPHENOL	95-48-7	1.5	USEPA RBRSL	0.5	0.33	0.25	0.2
2-NITROANILINE	88-74-4	0.15	USEPA RBRSL	0.05	0.82	0.62	0.075
2-NITROPHENOL	88-75-5	NC	None	NC	0.33	0.25	0.167
3,3'-DICHLOROBENZIDINE	91-94-1	0.00098	USEPA RBRSL	0.0003	0.33	0.25	0.114
3-NITROANILINE	99-09-2	0.01	FDEP LSCTL	7	0.82	0.62	0.094
4,6-DINITRO-2-METHYLPHENOL	534-52-1	0.005	USEPA RBRSL	0.002	0.82	0.62	0.337
4-BROMOPHENYL PHENYL ETHER	101-55-3	NC	None	NC	0.33	0.25	0.085
4-CHLORO-3-METHYLPHENOL	59-50-7	4.3	USEPA RBRSL	1	0.33	0.25	0.166
4-CHLOROANILINE	106-47-8	0.00014	USEPA RBRSL	0.00005	0.33	0.25	0.119
4-CHLOROPHENYL PHENYL ETHER	7005-72-3	NC	None	NC	0.33	0.25	0.078
4-METHYLPHENOL	106-44-5	0.15	USEPA RBRSL	0.05	0.33	0.25	0.187
4-NITROANILINE	100-01-6	0.0014	USEPA RBRSL	0.0005	0.82	0.62	0.134
4-NITROPHENOL	100-02-7	0.3	FDEP LSCTL	190	0.82	0.62	0.309
ACENAPHTHENE (1)	83-32-9	22	USEPA RBRSL	7	0.02	0.01	0.0015
ACENAPHTHYLENE (1)	208-96-8	22	USEPA RBRSL	7	0.02	0.01	0.0012
ACETOPHENONE	98-86-2	1.1	USEPA RBRSL	0.4	0.33	0.25	0.178
ANTHRACENE (1)	120-12-7	360	USEPA RBRSL	120	0.02	0.01	0.0012
ATRAZINE	1912-24-9	0.00019	USEPA RBRSL	0.00006	0.33	0.25	0.091
BENZALDEHYDE	100-52-7	0.81	USEPA RBRSL	0.3	0.33	0.25	0.12
BENZO(A)ANTHRACENE (1)	56-55-3	0.01	USEPA RBRSL	0.003	0.02	0.01	0.0019
BENZO(A)PYRENE (1)	50-32-8	0.0035	USEPA RBRSL	0.001	0.02	0.01	0.0033
BENZO(B)FLUORANTHENE (1)	205-99-2	0.035	USEPA RBRSL	0.01	0.02	0.01	0.0024
BENZO(G,H,I)PERYLENE (1)	191-24-2	120	USEPA RBRSL	40	0.02	0.01	0.002
BENZO(K)FLUORANTHENE (1)	207-08-9	0.35	USEPA RBRSL	0.1	0.02	0.01	0.0024

Analyte	CAS Number	PSL (mg/kg)	PSL Reference	Project Quantitation Limit Goal (mg/kg)	Katahdin		
					LOQ (mg/kg)	LOD (mg/kg)	MDL (mg/kg)
BIS(2-CHLOROETHOXY)METHANE	111-91-1	0.025	USEPA RBRSL	0.008	0.33	0.25	0.096
BIS(2-CHLOROETHYL)ETHER	111-44-4	0.000031	USEPA RBRSL	0.00001	0.33	0.25	0.081
BIS(2-CHLOROISOPROPYL)ETHER	39638-32-9	0.009	FDEP LSCTL	2	0.33	0.25	0.089
BIS(2-ETHYLHEXYL)PHTHALATE	117-81-7	1.1	USEPA RBRSL	0.4	0.33	0.25	0.098
BUTYL BENZYL PHTHALATE	85-68-7	0.51	USEPA RBRSL	0.2	0.33	0.25	0.093
CAPROLACTAM	105-60-2	4.5	USEPA RBRSL	2	0.33	0.25	0.144
CARBAZOLE	86-74-8	0.2	FDEP RSCTL	16	0.33	0.25	0.111
CHRYSENE (1)	218-01-9	1.1	USEPA RBRSL	0.4	0.02	0.01	0.0017
DIBENZO(A,H)ANTHRACENE (1)	53-70-3	0.011	USEPA RBRSL	0.004	0.02	0.01	0.0018
DIBENZOFURAN	132-64-9	0.68	USEPA RBRSL	0.2	0.33	0.25	0.079
DIETHYL PHTHALATE	84-66-2	12	USEPA RBRSL	4	0.33	0.25	0.08
DIMETHYL PHTHALATE	131-11-3	380	FDEP LSCTL	230,000	0.33	0.25	0.078
DI-N-BUTYL PHTHALATE	84-74-2	9.2	USEPA RBRSL	3	0.33	0.25	0.101
DI-N-OCTYL PHTHALATE	117-84-0	1,700	FDEP RSCTL	570	0.33	0.25	0.211
FLUORANTHENE (1)	206-44-0	160	USEPA RBRSL	53	0.02	0.01	0.0018
FLUORENE (1)	86-73-7	27	USEPA RBRSL	9	0.02	0.01	0.0032
HEXACHLOROBENZENE	118-74-1	0.00053	USEPA RBRSL	0.0002	0.33	0.25	0.082
HEXACHLOROBUTADIENE	87-68-3	0.0017	USEPA RBRSL	0.0006	0.33	0.25	0.083
HEXACHLOROCYCLOPENTADIENE	77-47-4	0.68	USEPA RBRSL	0.2	0.33	0.25	0.082
HEXACHLOROETHANE	67-72-1	0.0029	USEPA RBRSL	0.001	0.33	0.25	0.096
INDENO(1,2,3-CD)PYRENE (1)	193-39-5	0.12	USEPA RBRSL	0.04	0.02	0.01	0.0019
ISOPHORONE	78-59-1	0.023	USEPA RBRSL	0.008	0.33	0.25	0.075
NAPHTHALENE (1)	91-20-3	0.00047	USEPA RBRSL	0.0002	0.02	0.01	0.0026
NITROBENZENE	98-95-3	0.000079	USEPA RBRSL	0.00003	0.33	0.25	0.091
N-NITROSO-DI-N-PROPYLAMINE	621-64-7	0.0000072	USEPA RBRSL	0.000002	0.33	0.25	0.083
N-NITROSODIPHENYLAMINE	86-30-6	0.075	USEPA RBRSL	0.03	0.33	0.25	0.219
PENTACHLOROPHENOL	87-86-5	0.0057	USEPA RBRSL	0.002	0.82	0.62	0.237
PHENANTHRENE (1)	85-01-8	120	USEPA RBRSL	40	0.02	0.01	0.0018
PHENOL	108-95-2	6.3	USEPA RBRSL	2	0.33	0.25	0.156
PYRENE (1)	129-00-0	120	USEPA RBRSL	40	0.02	0.01	0.0021

(1) 8270D Selected Ion Monitoring (SIM) SOP will be utilized for low level PAHs.

Project Screening Level Reference: JAX SS BKS (NAS Jacksonville surface soil background value); JAX SB BKG (NAS Jacksonville subsurface soil background value) (ABB-ES, 1996); USEPA RBRSL (USEPA Regions 3, 6, and 9 Risk-based Regional Screening Level - Protection of Groundwater); USEPA RSL (USEPA Regions 3, 6, and 9 Residential Regional Screening Level); FDEP LSCTL (FDEP Chapter 62-777, F.A.C Soil Cleanup Target Levels, Leachability based on Groundwater Criteria); FDEP RSCTL (FDEP Chapter 62-777, F.A. C., Soil Cleanup Target Levels, Resident Direct Exposure levels).

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Bolded compounds indicate that the PSL is between the laboratory LOQ and LOD. The Partnering Team has agreed to accept this data for decision making if results less than the LOQ are "J" qualified and their impact on the attainment of project objectives will be described in the uncertainties section of the Risk Assessment**.**

Shaded and Bold compounds indicate the PSL is less than the LOD. The Partnering Team has agreed to replace the PSLs with the laboratory LOQs for decision making purposes, as suggested in "Guidance for the Selection of Analytical Methods for the Evaluation of Practical Quantitation Limits" (FDEP, 2004).

Matrix: Soil
Analytical Group: TRPH

Analyte	CAS Number	PSL (mg/kg)	PSL Reference	Project Quantitation Limit Goal (mg/kg)	Katahdin		
					LOQ (mg/kg)	LOD (mg/kg)	MDL (mg/kg)
TPH (C08-C40)	NA	340	FDEP LSCTL	0.2	5	4	2.6

Project Screening Level Reference: JAX SS BKS (NAS Jacksonville surface soil background value); JAX SB BKG (NAS Jacksonville subsurface soil background value) (ABB-ES, 1996); USEPA RBRSL (USEPA Regions 3, 6, and 9 Risk-based Regional Screening Level - Protection of Groundwater); USEPA RSL (USEPA Regions 3, 6, and 9 Residential Regional Screening Level); FDEP LSCTL (FDEP Chapter 62-777, F.A.C Soil Cleanup Target Levels, Leachability based on Groundwater Criteria.

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Shaded and Bold compounds indicate the PSL is less than the LOD. The Partnering Team has agreed to replace the PSLs with the laboratory LOQs for decision making purposes, as suggested in "Guidance for the Selection of Analytical Methods for the Evaluation of Practical Quantitation Limits" (FDEP, 2004).

Matrix: Groundwater
Analytical Group: Metals

Analyte	CAS Number	PSL (ug/L)	PSL Reference	Project Quantitation Limit Goal (ug/L)	Katahdin		
					LOQ (ug/L)	LOD (ug/L)	MDL (ug/L)
ALUMINUM	7429-90-5	200	FL GCTL	66.7	300	100	15
ANTIMONY	7440-36-0	6	FL GCTL	2	8	5	1.5
ARSENIC	7440-38-2	0.045	USEPA TAPWATER	0.015	8	5	1.9
BARIUM	7440-39-3	2,000	FL GCTL	667	5	3	0.44
BERYLLIUM	7440-41-7	4	FL GCTL	1.3	5	0.5	0.042
CADMIUM	7440-43-9	5	FL GCTL	1.7	10	0.5	0.04
CALCIUM	7440-70-2	NC	None	NC	100	80	5.8
CHROMIUM	7440-47-3	100	FL GCTL	33.3	15	4	0.32
COBALT	7440-48-4	11	USEPA TAPWATER	3.7	30	4	0.28
COPPER	7440-50-8	1,000	FL GCTL	333	25	10	0.48
IRON	7439-89-6	300	FL GCTL	100	100	80	6.3
LEAD	7439-92-1	15	FL GCTL	5	5	4	0.73
MAGNESIUM	7439-95-4	NC	None	NC	100	80	4.8
MANGANESE	7439-96-5	50	FL GCTL	16.7	5	4	0.37
MERCURY	7439-97-6	0.57	USEPA TAPWATER	0.19	0.2	0.1	0.037
NICKEL	7440-02-0	100	FL GCTL	33.3	40	4	0.29
POTASSIUM	7440-09-7	NC	None	NC	1,000	500	105
SELENIUM	7782-49-2	50	FL GCTL	16.7	10	7	3.7
SILVER	7440-22-4	100	FL GCTL	33.3	15	4	0.48
SODIUM	7440-23-5	160,000	FL GCTL	53,300	1,000	500	34
THALLIUM	7440-28-0	2	FL GCTL	0.67	15	5	0.67
VANADIUM	7440-62-2	49	FL GCTL	16.3	25	4	0.39
ZINC	7440-66-6	5,000	FL GCTL	1670	25	10	0.51

µg/L = micrograms per liter

Project Screening Level Reference: USEPA TAPWATER (USEPA Regions 3, 6, and 9 RSLs for Tapwater), FL GCTL (Chapter 62-777, F.A.C., Groundwater Cleanup Target Levels).

Bolded compounds indicate that the PSL is between the laboratory LOQ and LOD. The Partnering Team has agreed to accept this data for decision making if results less than the LOQ are "J" qualified and their impact on the attainment of project objectives will be described in the uncertainties section of the Risk Assessment.

Shaded and Bold compounds indicate the PSL is less than the LOD. The Partnering Team has agreed to replace the PSLs with the laboratory LOQs for decision making purposes, as suggested in “Guidance for the Selection of Analytical Methods for the Evaluation of Practical Quantitation Limits” (FDEP, 2004).

Matrix: Groundwater
Analytical Group: PCBs

Analyte	CAS Number	PSL (ug/L)	PSL Reference	Project Quantitation Limit Goal (ug/L)	Katahdin		
					LOQ (ug/L)	LOD (ug/L)	MDL (ug/L)
AROCLOR-1016	12674-11-2	0.96	USEPA TAPWATER	0.32	0.1	0.05	0.03
AROCLOR-1221	11104-28-2	0.0068	USEPA TAPWATER	0.0023	0.1	0.05	0.04
AROCLOR-1232	11141-16-5	0.0068	USEPA TAPWATER	0.0023	0.1	0.05	0.0178
AROCLOR-1242	53469-21-9	0.034	USEPA TAPWATER	0.011	0.1	0.05	0.036
AROCLOR-1248	12672-29-6	0.034	USEPA TAPWATER	0.011	0.1	0.05	0.04
AROCLOR-1254	11097-69-1	0.034	USEPA TAPWATER	0.011	0.1	0.05	0.0164
AROCLOR-1260	11096-82-5	0.034	USEPA TAPWATER	0.011	0.1	0.05	0.034
TOTAL AROCLOR	1336-36-3	0.5	FL GCTL	0.17	1	1	1

1 Laboratory reports individual Aroclor values.

Project Screening Level Reference: USEPA TAPWATER (USEPA Regions 3, 6, and 9 RSLs for Tapwater), FL GCTL (Chapter 62-777, F.A.C., Groundwater Cleanup Target Levels).

Bolded compounds indicate that the PSL is between the laboratory LOQ and LOD. The Partnering Team has agreed to accept this data for decision making if results less than the LOQ are "J" qualified and their impact on the attainment of project objectives will be described in the uncertainties section of the Risk Assessment.

Shaded and Bold compounds indicate the PSL is less than the LOD. The Partnering Team has agreed to replace the PSLs with the laboratory LOQs for decision making purposes, as suggested in "Guidance for the Selection of Analytical Methods for the Evaluation of Practical Quantitation Limits" (FDEP, 2004).

Matrix: Groundwater
Analytical Group: VOCs

Analyte	CAS Number	PSL (ug/L)	PSL Reference	Project Quantitation Limit Goal (ug/L)	Katahdin		
					LOQ (ug/L)	LOD (ug/L)	MDL (ug/L)
1,1,1-TRICHLOROETHANE	71-55-6	200	FL GCTL	67	1	0.5	0.2
1,1,2,2-TETRACHLOROETHANE	79-34-5	0.067	USEPA TAPWATER	0.02	1	0.5	0.38
1,1,2-TRICHLOROETHANE	79-00-5	0.24	USEPA TAPWATER	0.08	1	0.5	0.33
1,1,2-TRICHLOROTRIFLUOROETHANE	76-13-1	59,000	USEPA TAPWATER	20,000	1	0.5	0.31
1,1-DICHLOROETHANE	75-34-3	2.4	USEPA TAPWATER	0.8	1	0.5	0.21
1,1-DICHLOROETHENE	75-35-4	7	FL GCTL	2	1	0.5	0.35
1,2,4-TRICHLOROBENZENE	120-82-1	2.3	USEPA TAPWATER	0.8	1	0.5	0.37
1,2-DIBROMO-3-CHLOROPROPANE	96-12-8	0.00032	USEPA TAPWATER	0.0001	1	0.5	0.5
1,2-DIBROMOETHANE	106-93-4	0.0065	USEPA TAPWATER	0.002	1	0.5	0.22
1,2-DICHLOROBENZENE	95-50-1	370	USEPA TAPWATER	120	1	0.5	0.15
1,2-DICHLOROETHANE	107-06-2	0.15	USEPA TAPWATER	0.05	1	0.5	0.2
1,2-DICHLOROPROPANE	78-87-5	0.39	USEPA TAPWATER	0.1	1	0.5	0.25
1,3-DICHLOROBENZENE	541-73-1	210	FL GCTL	70	1	0.5	0.26
1,4-DICHLOROBENZENE	106-46-7	0.43	USEPA TAPWATER	0.1	1	0.5	0.24
2-BUTANONE	78-93-3	4,200	FL GCTL	1,400	5	2.5	1.31
2-HEXANONE	591-78-6	47	USEPA TAPWATER	16	5	2.5	1.7
4-METHYL-2-PENTANONE	108-10-1	560	FL GCTL	190	5	2.5	1.32
ACETONE	67-64-1	6,300	FL GCTL	2,100	5	2.5	2.21
BENZENE	71-43-2	0.41	USEPA TAPWATER	0.1	1	0.5	0.26
BROMODICHLOROMETHANE	75-27-4	0.12	USEPA TAPWATER	0.04	1	0.5	0.33
BROMOFORM	75-25-2	4.4	FL GCTL	1	1	0.5	0.23
BROMOMETHANE	74-83-9	8.7	USEPA TAPWATER	3	2	1	0.49
CARBON DISULFIDE	75-15-0	700	FL GCTL	230	1	0.5	0.25
CARBON TETRACHLORIDE	56-23-5	0.44	USEPA TAPWATER	0.1	1	0.5	0.22
CHLOROBENZENE	108-90-7	91	USEPA TAPWATER	30	1	0.5	0.22
CHLORODIBROMOMETHANE	124-48-1	0.15	USEPA TAPWATER	0.05	1	0.5	0.3
CHLOROETHANE	75-00-3	12	FL GCTL	4	2	1	0.55
CHLOROFORM	67-66-3	0.19	USEPA TAPWATER	0.06	1	0.5	0.32
CHLOROMETHANE	74-87-3	2.7	FL GCTL	0.9	2	1	0.36
CIS-1,2-DICHLOROETHENE	156-59-2	70	FL GCTL	23	1	0.5	0.21
CIS-1,3-DICHLOROPROPENE	10061-01-5	0.43	USEPA TAPWATER	0.1	1	0.5	0.19
CYCLOHEXANE	110-82-7	13,000	USEPA TAPWATER	4,300	1	0.5	0.31
DICHLORODIFLUOROMETHANE	75-71-8	390	USEPA TAPWATER	130	2	1	0.24
ETHYLBENZENE	100-41-4	1.5	USEPA TAPWATER	0.5	1	0.5	0.21
ISOPROPYLBENZENE	98-82-8	0.8	FL GCTL	0.3	1	0.5	0.23

Analyte	CAS Number	PSL (ug/L)	PSL Reference	Project Quantitation Limit Goal (ug/L)	Katahdin		
					LOQ (ug/L)	LOD (ug/L)	MDL (ug/L)
METHYL ACETATE	79-20-9	3,000	FL GCTL	1,000	1	0.5	0.53
METHYL CYCLOHEXANE	108-87-2	NC	None	NC	1	0.5	0.3
METHYL TERT-BUTYL ETHER	1634-04-4	12	USEPA TAPWATER	4	1	0.5	0.36
METHYLENE CHLORIDE	75-09-2	4.8	USEPA TAPWATER	2	5	2.5	1.13
STYRENE	100-42-5	100	FL GCTL	33	1	0.5	0.23
TETRACHLOROETHENE	127-18-4	0.11	USEPA TAPWATER	0.04	1	0.5	0.4
TOLUENE	108-88-3	40	FL GCTL	13	1	0.5	0.27
TOTAL XYLENES	1330-20-7	20	FL GCTL	7	3	1.5	0.25
TRANS-1,2-DICHLOROETHENE	156-60-5	100	FL GCTL	33	1	0.5	0.25
TRANS-1,3-DICHLOROPROPENE	10061-02-6	NC	None	NC	1	0.5	0.2
TRICHLOROETHENE	79-01-6	2	USEPA TAPWATER	0.7	1	0.5	0.28
TRICHLOROFLUOROMETHANE	75-69-4	1,300	USEPA TAPWATER	430	2	1	0.24
VINYL CHLORIDE	75-01-4	0.016	USEPA TAPWATER	0.005	2	1	0.25

Project Screening Level Reference: USEPA TAPWATER (USEPA Regions 3, 6, and 9 RSLs for Tapwater), FL GCTL (Chapter 62-777, F.A.C., Groundwater Cleanup Target Levels).

Bolded compounds indicate that the PSL is between the laboratory LOQ and LOD. The Partnering Team has agreed to accept this data for decision making if results less than the LOQ are "J" qualified and their impact on the attainment of project objectives will be described in the uncertainties section of the Risk Assessment.

Shaded and Bold compounds indicate the PSL is less than the LOD. The Partnering Team has agreed to replace the PSLs with the laboratory LOQs for decision making purposes, as suggested in "Guidance for the Selection of Analytical Methods for the Evaluation of Practical Quantitation Limits" (FDEP, 2004).

Matrix: Groundwater
Analytical Group: SVOCs (including Low Level PAHs by SIM [1])

Analyte	CAS Number	PSL (ug/L)	PSL Reference	Project Quantitation Limit Goal (ug/L)	Katahdin		
					LOQ (ug/L)	LOD (ug/L)	MDL (ug/L)
1,1-BIPHENYL	92-52-4	0.5	FL GCTL	0.2	10	7.5	2.7
2,4,5-TRICHLOROPHENOL	95-95-4	1	FL GCTL	0.3	25	18.75	3.6
2,4,6-TRICHLOROPHENOL	88-06-2	3.2	FL GCTL	1	10	7.5	2.7
2,4-DICHLOROPHENOL	120-83-2	0.3	FL GCTL	0.1	10	7.5	3
2,4-DIMETHYLPHENOL	105-67-9	140	FL GCTL	47	10	7.5	4.4
2,4-DINITROPHENOL	51-28-5	14	FL GCTL	5	25	18.75	1
2,4-DINITROTOLUENE	121-14-2	0.05	FL GCTL	0.02	10	7.5	2.2
2,6-DINITROTOLUENE	606-20-2	0.05	FL GCTL	0.02	10	7.5	2
2-CHLORONAPHTHALENE	91-58-7	560	FL GCTL	190	10	7.5	2.9
2-CHLOROPHENOL	95-57-8	35	FL GCTL	12	10	0.1	3.2
2-METHYLNAPHTHALENE (1)	91-57-6	28	FL GCTL	9	0.2	0.1	0.077
2-METHYLPHENOL	95-48-7	35	FL GCTL	12	10	7.5	3.8
2-NITROANILINE	88-74-4	21	FL GCTL	7	2	18.75	1.8
2-NITROPHENOL	88-75-5	NC	None	NC	10	7.5	2.7
3,3'-DICHLOROBENZIDINE	91-94-1	0.08	FL GCTL	0.03	10	7.5	1.1
3-NITROANILINE	99-09-2	1.7	FL GCTL	0.6	25	18.75	1.5
4,6-DINITRO-2-METHYLPHENOL	534-52-1	2.9	USEPA TAPWATER	1	25	18.75	2
4-BROMOPHENYL PHENYL ETHER	101-55-3	NC	None	NC	10	7.5	1.9
4-CHLORO-3-METHYLPHENOL	59-50-7	63	FL GCTL	21	10	7.5	3.6
4-CHLOROANILINE	106-47-8	0.34	USEPA TAPWATER	0.1	10	7.5	1.9
4-CHLOROPHENYL PHENYL ETHER	7005-72-3	NC	None	NC	10	7.5	2.2
4-METHYLPHENOL	106-44-5	3.5	FL GCTL	1	10	7.5	5.6
4-NITROANILINE	100-01-6	1.7	FL GCTL	0.6	25	18.75	1.6
4-NITROPHENOL	100-02-7	56	FL GCTL	19	25	18.75	1.8
ACENAPHTHENE (1)	83-32-9	20	FL GCTL	7	0.2	0.1	0.064
ACENAPHTHYLENE (1)	208-96-8	210	FL GCTL	70	0.2	0.1	0.054
ACETOPHENONE	98-86-2	700	FL GCTL	230	10	7.5	3.9
ANTHRACENE (1)	120-12-7	2,100	FL GCTL	700	0.2	0.1	0.044
ATRAZINE	1912-24-9	0.29	USEPA TAPWATER	0.1	10	7.5	3.3
BENZALDEHYDE	100-52-7	700	FL GCTL	230	10	7.5	1
BENZO(A)ANTHRACENE (1)	56-55-3	0.029	USEPA TAPWATER	0.01	0.2	0.1	0.046
BENZO(A)PYRENE (1)	50-32-8	0.0029	USEPA TAPWATER	0.001	0.2	0.1	0.066
BENZO(B)FLUORANTHENE (1)	205-99-2	0.029	USEPA TAPWATER	0.01	0.2	0.1	0.089
BENZO(G,H,I)PERYLENE (1)	191-24-2	210	FL GCTL	70	0.2	0.1	0.065
BENZO(K)FLUORANTHENE (1)	207-08-9	0.29	USEPA TAPWATER	0.1	0.2	0.1	0.049

Analyte	CAS Number	PSL (ug/L)	PSL Reference	Project Quantitation Limit Goal (ug/L)	Katahdin		
					LOQ (ug/L)	LOD (ug/L)	MDL (ug/L)
BIS(2-CHLOROETHOXY)METHANE	111-91-1	110	USEPA TAPWATER	37	10	7.5	2.1
BIS(2-CHLOROETHYL)ETHER	111-44-4	0.012	USEPA TAPWATER	0.004	10	7.5	2
BIS(2-CHLOROISOPROPYL)ETHER	39638-32-9	0.5	FL GCTL	0.2	10	7.5	2.1
BIS(2-ETHYLHEXYL)PHTHALATE	117-81-7	4.8	USEPA TAPWATER	2	10	7.5	1.8
BUTYL BENZYL PHTHALATE	85-68-7	35	USEPA TAPWATER	12	10	7.5	1.9
CAPROLACTAM	105-60-2	18,000	USEPA TAPWATER	6,000	10	7.5	0.4
CARBAZOLE	86-74-8	1.8	FL GCTL	0.6	10	7.5	2.1
CHRYSENE (1)	218-01-9	2.9	USEPA TAPWATER	1	0.2	0.1	0.036
DIBENZO(A,H)ANTHRACENE (1)	53-70-3	0.0029	USEPA TAPWATER	0.001	0.2	0.1	0.07
DIBENZOFURAN	132-64-9	28	FL GCTL	9	10	7.5	1.6
DIETHYL PHTHALATE	84-66-2	5,600	FL GCTL	1,900	10	7.5	2
DIMETHYL PHTHALATE	131-11-3	70,000	FL GCTL	23,000	10	7.5	2
DI-N-BUTYL PHTHALATE	84-74-2	700	FL GCTL	230	10	7.5	2.5
DI-N-OCTYL PHTHALATE	117-84-0	140	FL GCTL	47	10	7.5	1.8
FLUORANTHENE (1)	206-44-0	280	FL GCTL	93	0.2	0.1	0.073
FLUORENE (1)	86-73-7	280	FL GCTL	93	0.2	0.1	0.061
HEXACHLOROBENZENE	118-74-1	0.042	USEPA TAPWATER	0.01	10	7.5	2.1
HEXACHLOROBUTADIENE	87-68-3	0.4	FL GCTL	0.1	10	7.5	1.8
HEXACHLOROCYCLOPENTADIENE	77-47-4	50	FL GCTL	17	10	7.5	1.2
HEXACHLOROETHANE	67-72-1	2.5	FL GCTL	0.8	10	7.5	2.3
INDENO(1,2,3-CD)PYRENE (1)	193-39-5	0.029	USEPA TAPWATER	0.01	0.2	0.1	0.052
ISOPHORONE	78-59-1	37	FL GCTL	12	10	7.5	1.7
NAPHTHALENE (1)	91-20-3	0.14	USEPA TAPWATER	0.05	0.2	0.1	0.064
NITROBENZENE	98-95-3	0.12	USEPA TAPWATER	0.04	10	7.5	3.1
N-NITROSO-DI-N-PROPYLAMINE	621-64-7	0.005	FL GCTL	0.002	10	7.5	1.9
N-NITROSODIPHENYLAMINE	86-30-6	7.1	FL GCTL	2	10	7.5	3.7
PENTACHLOROPHENOL	87-86-5	0.56	USEPA TAPWATER	0.2	25	18.75	2.3
PHENANTHRENE (1)	85-01-8	210	FL GCTL	70	0.2	0.1	0.051
PHENOL	108-95-2	10	FL GCTL	3	10	7.5	1.8
PYRENE (1)	129-00-0	210	FL GCTL	70	0.2	0.1	0.059

(1) 8270D SIM SOP will be utilized for low level PAHs.

Project Screening Level Reference: USEPA TAPWATER (USEPA Regions 3, 6, and 9 RSLs for Tapwater), FL GCTL (Chapter 62-777, F.A.C., Groundwater Cleanup Target Levels).

Bolded compounds indicate that the PSL is between the laboratory LOQ and LOD. The Partnering Team has agreed to accept this data for decision making if results less than the LOQ are "J" qualified and their impact on the attainment of project objectives will be described in the uncertainties section of the Risk Assessment.

Shaded and Bold compounds indicate the PSL is less than the LOD. The Partnering Team has agreed to replace the PSLs with the laboratory LOQs for decision making purposes, as suggested in "Guidance for the Selection of Analytical Methods for the Evaluation of Practical Quantitation Limits" (FDEP, 2004).

Matrix: Groundwater
Analytical Group: TRPH

Analyte	CAS Number	Project Screening Levels (ug/L)	Project Screening Level Reference	Project Quantitation Limit Goal (ug/L)	Katahdin		
					LOQ (ug/L)	LOD (ug/L)	MDL (ug/L)
TPH (C08-C40)	NA	5,000	FL GCTL	1,700	75	38	9.1

Project Screening Level Reference: USEPA TAPWATER (USEPA Regions 3, 6, and 9 RSLs for Tapwater), FL GCTL (Chapter 62-777, F.A.C., Groundwater Cleanup Target Levels).

SAP Worksheet #16 – Project Schedule / Timeline Table
(UFP-QAPP Manual Section 2.8.2)

Activities	Organization	Dates (MM/DD/YY)		Deliverable	Deliverable Due Date
		Anticipated Date(s) of Initiation	Anticipated Date of Completion		
Field Team Mobilization	Tetra Tech	05/04/11	05/05/11	NA	NA
Phase I Soil, Well Installations and Groundwater Sampling	Tetra Tech	05/05/11	05/13/11	Team Data Presentation	7/19/11
Phase II Ground Water Investigation	Tetra Tech	06/27/12	07/4/11	Team Data Presentation	10/11/11
Phase II Well Installations	Tetra Tech	07/11/11	07/29/11	Team Data Presentation	1/10/12
Demobilization	Tetra Tech	08/01/11	08/01/11	NA	NA
RI Report Draft Submittal	Tetra Tech	08/01/11	09/29/11	Report Draft Submittal	09/30/11

SAP Worksheet #17 – Sampling Design and Rationale
(UFP-QAPP Manual Section 3.1.1)

The proposed groundwater and soil sampling locations for PSC 45 were chosen based on the current understanding of site-specific conditions and the need to collect data that will help to delineate contamination in soil and groundwater and evaluate human health risks as described in Worksheet #11.

Mobilization will occur in the spring of 2011. The groundwater and soil sampling activities will occur within a 20-day period. Demobilization will occur when the field crew leaves the site on or before the 21st day of field activity (see Worksheet #16).

The SOPs are listed in Worksheet #14. Activities at PSC 45 will follow FDEP SOP FS 2200 for groundwater sampling and FDEP SOP FS 3000 for soil samples (FDEP, 2008).

17.1 SOIL SAMPLING

The target analytes associated with the soil samples are presented in Worksheet #15. The Analytical Method/SOPs are identified in Worksheet #23. The number of QC samples is presented in Worksheet #20.

Soil samples from 10 locations will be collected from one soil horizon (see Worksheet #11, Worksheet #18, and Figure 17-1). The sampling intervals will be from zero (0) to six (6) inches, to approximately the top of the water table plus 2 feet. This approach yields 10 soil samples. Field QC samples will be collected including a minimum of one Field Duplicate, one sample for Matrix Spike/Matrix Spike Duplicate (MS/MSD) analysis (or MS/sample duplicate for metals), and one Equipment Rinsate Blank.

17.2 MONITORING WELL GROUNDWATER SAMPLING

The monitoring wells will be developed to remove fine sediment from around the screened interval of the well. Wells will be developed by pumping using either a submersible pump or peristaltic pump. Field parameters (pH, temperature, turbidity, and specific conductance) will be measured at equally-spaced time intervals during well development. Wells will be developed a maximum of one hour or until the field measurements become stable and the development water is visibly clear. Water quality stabilization will be determined using the following criteria:

- Temperature $\pm$ 0.2 degrees ºC
- pH $\pm$ 0.2 standard units

- Specific conductivity ± 5 percent of reading

The data will be recorded on a monitoring well development record (see Appendix C). No sooner than 24 hours after development, groundwater samples will be collected from monitoring wells in general accordance with to FDEP SOP 001/01 FS2200: Groundwater Sampling and FS1000. Prior to obtaining samples, synoptic water levels and total well depths will be measured and recorded on a groundwater level measurement sheet (see Appendix C). A second round of water levels will be collected no sooner than one month later and submitted on a separate field data sheet.

The wells will be purged using a peristaltic pump using low flow quiescent purging techniques per FDEP SOPs. The data will be recorded on a low flow purge data sheet (see Appendix C). Depending on the groundwater parameters, up to five well volumes may be purged. If wells are purged dry with less than one well volume removed, the water level in the well will be allowed to recover enough to collect three field readings (pH, temperature, turbidity, dissolved oxygen, and specific conductance) prior to collecting a water sample. If the well does not purge dry using the low flow purging technique, groundwater characteristics will be taken (per FDEP SOP FS 2210) after each well volume of water is purged, or at 2- to 10-minute intervals, depending on the flow rate. Stabilization will be defined according to the following scenarios:

3) When purging a well that has a partially submerged well screen, a minimum of one well volume will be purged prior to collecting measurements of field parameters listed below. If the well screen is fully submerged, then a minimum of one volume of the pump, associated tubing, and flow cell will be purged prior to collecting field parameters listed below. Purging will be considered complete when three consecutive measurements in which the field parameters are within the desired limits as shown below.

    - Temperature ± 0.2° C
    - pH ± 0.2 Standard Units
    - Specific Conductivity ± 5 percent of reading
    - Dissolved oxygen is not greater than 20 percent of saturation at the field measured temperature
    - Turbidity is not greater than 20 NTUs

4) When purging a well and the stabilization described in 1) above cannot be achieved, three consecutive measurements of the following parameters are required:

    - Dissolved oxygen ± 0.2 mg/L or 10 percent, whichever is greater
    - Temperature ± 0.2° C
    - pH ± 0.2 Standard Units.
    - Specific Conductivity ± 5 percent of reading

- Turbidity ± 5 NTUs or 10 percent, whichever is greater

If stabilization is not achieved, five screen volumes must be removed prior to samples being collected in the appropriate sample containers. Samples to be analyzed for volatile constituents will be collected first and immediately sealed in 40-milliliter vials so that no headspace exists. The data acquired during sampling will be recorded on a groundwater sample log sheet (see Appendix C).

Since data obtained from groundwater during the SA did not include all constituents detected in soils, a shallow groundwater monitoring well will be installed at the immediate downgradient edge of the former disposal pit for the purpose of determining if groundwater is impacted as a result of contamination bound up in shallow soils. The monitoring well will be a 2-inch inside diameter, Schedule 40 flush-joint PVC riser and flush-joint 0.010-inch factory-slotted well screen, installed using DPT techniques. The monitoring well will be developed and sampled for the full range of constituents analyzed in soil including metal, PCB, TRPH, VOC, and SVOC constituents as show in Worksheet #18.

17.2 DPT GROUNDWATER SAMPLING

The target analytes associated with the DPT groundwater samples will be determined after completion of Phase I. The appropriate analyses as determined from Phase I will be selected from the groundwater monitoring analyses that are presented in Worksheet #15. The Analytical Method/SOPs are identified in Worksheet #23 will be used for the appropriate analyses. The number of QC samples is presented in Worksheet #20.

Groundwater samples, at each of 7 borings installed by DPT (see Worksheet #11, Worksheet #18, and Figure 17-2) will be collected from 4 intervals, yielding a total of 28 groundwater samples. Field QC samples will be collected, including 2 samples for MS/MSD analysis (or MS/sample duplicate for metals), 3 VOC Trip Blanks, and 2 Equipment Rinsate Blanks.

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Legend

- Proposed Soil Boring Location
- Phase I Soil Boring Location
- OU 3 NW Boundary

DRAWN BY / DATE	T.WHEATON 07/08/10
CHECKED BY / DATE	B. PEEBLES 09/21/10
COST/SCHED AREA	
SCALE	AS NOTED

PROPOSED SOIL BORING LOCATIONS
PSC 45 - BUILDING 200
NAS JACKSONVILLE
JACKSONVILLE, FLORIDA

CONTRACT NUMBER	CTO JM 19
APPROVED BY / DATE	____ ____
APPROVED BY / DATE	____ ____
FIGURE NO.	FIGURE 17-1
REV	0

P:\GIS\JACKSONVILLE_NAS\MXD\BUILDING200_PROP_DPT.MXD 09/21/10 TW

Legend

- Proposed DPT Location
- Phase II Groundwater Sample Location
- OU 3 NW Boundary

DRAWN BY / DATE	T.WHEATON 07/08/10
CHECKED BY / DATE	B. PEEBLES 09/21/10
COST/SCHED AREA	
SCALE	AS NOTED

PROPOSED DPT LOCATIONS
BUILDING 200
NAS JACKSONVILLE
JACKSONVILLE, FLORIDA

CONTRACT NUMBER	CTO JM 19
APPROVED BY / DATE	____ ____
APPROVED BY / DATE	____ ____
FIGURE NO.	FIGURE 17-2
REV	0

SAP Worksheet #18 – Sampling Locations and Methods/SOP Requirements Table
(UFP-QAPP Manual Section 3.1.1)

SAP Worksheet #18.1 –-Groundwater Sampling Locations and Methods/SOP Requirements Table

Sampling Location	Identification (ID) Number	Matrix	Depth (feet)[1]	Analytical Group	Number of Samples (identify field duplicates)	Sampling SOP Reference
GROUNDWATER SAMPLES DPT						
JAX45-DPT12	JAX45-DPT12-12-Date	Water	12-16	VOCs, SVOCs, Low Level PAHs, PCBs, Metals, and TRPH	1	FS 1000, FS 2000, and FS 2200
JAX45-DPT12	JAX45-DPT12-20-Date	Water	20- 24	VOCs, SVOCs, Low Level PAHs, PCBs, Metals, and TRPH	1	FS 1000, FS 2000, and FS 2200
JAX45-DPT12	JAX45-DPT12-40-Date	Water	40–44	VOCs, SVOCs, Low Level PAHs, PCBs, Metals, and TRPH	1 + duplicate	FS 1000, FS 2000, and FS 2200
JAX45-DPT12	JAX45-DPT12-60-Date	Water	60-64	VOCs, SVOCs, Low Level PAHs, PCBs, Metals, and TRPH	1	FS 1000, FS 2000, and FS 2200
JAX45-DPT13	JAX45-DPT13-12-Date	Water	12-16	VOCs, SVOCs, Low Level PAHs, PCBs, Metals, and TRPH	1	FS 1000, FS 2000, and FS 2200

Sampling Location	Identification (ID) Number	Matrix	Depth (feet)[1]	Analytical Group	Number of Samples (identify field duplicates)	Sampling SOP Reference
JAX45-DPT13	JAX45-DPT13-20-Date	Water	20- 24	VOCs, SVOCs, Low Level PAHs, PCBs, Metals, and TRPH	1	FS 1000, FS 2000, and FS 2200
JAX45-DPT13	JAX45-DPT13-40-Date	Water	40–44	VOCs, SVOCs, Low Level PAHs, PCBs, Metals, and TRPH	1	FS 1000, FS 2000, and FS 2200
JAX45-DPT13	JAX45-DPT13-60-Date	Water	60-64	VOCs, SVOCs, Low Level PAHs, PCBs, Metals, and TRPH	1	FS 1000, FS 2000, and FS 2200
JAX45-DPT14	JAX45-DPT14-12-Date	Water	12-16	VOCs, SVOCs, Low Level PAHs, PCBs, Metals, and TRPH	1	FS 1000, FS 2000, and FS 2200
JAX45-DPT14	JAX45-DPT14-20-Date	Water	20- 24	VOCs, SVOCs, Low Level PAHs, PCBs, Metals, and TRPH	1	FS 1000, FS 2000, and FS 2200
JAX45-DPT14	JAX45-DPT14-40-Date	Water	40–44	VOCs, SVOCs, Low Level PAHs, PCBs, Metals, and TRPH	1	FS 1000,FS 2000, and FS 2200
JAX45-DPT14	JAX45-DPT14-60-Date	Water	60-64	VOCs, SVOCs, Low Level PAHs, PCBs, Metals, and TRPH	1	FS 1000,FS 2000, and FS 2200

Sampling Location	Identification (ID) Number	Matrix	Depth (feet)[1]	Analytical Group	Number of Samples (identify field duplicates)	Sampling SOP Reference
JAX45-DPT15	JAX45-DPT15-12-Date	Water	12-16	VOCs, SVOCs, Low Level PAHs, PCBs, Metals, and TRPH	1	FS 1000,FS 2000, and FS 2200
JAX45-DPT15	JAX45-DPT15-20-Date	Water	20- 24	VOCs, SVOCs, Low Level PAHs, PCBs, Metals, and TRPH	1	FS 1000, FS 2000, and FS 2200
JAX45-DPT15	JAX45-DPT15-40-Date	Water	40–44	VOCs, SVOCs, Low Level PAHs, PCBs, Metals, and TRPH	1	FS 1000, FS 2000, and FS 2200
JAX45-DPT15	JAX45-DPT15-60-Date	Water	60-64	VOCs, SVOCs, Low Level PAHs, PCBs, Metals, and TRPH	1	FS 1000, FS 2000, and FS 2200
JAX45-DPT16	JAX45-DPT16-12-Date	Water	12-16	VOCs, SVOCs, Low Level PAHs, PCBs, Metals, and TRPH	1	FS 1000, FS 2000, and FS 2200
JAX45-DPT16	JAX45-DPT16-20-Date	Water	20- 24	VOCs, SVOCs, Low Level PAHs, PCBs, Metals, and TRPH	1	FS 1000, FS 2000, and FS 2200
JAX45-DPT16	JAX45-DPT16-40-Date	Water	40–44	VOCs, SVOCs, Low Level PAHs, PCBs, Metals, and TRPH	1	FS 1000, FS 2000, and FS 2200

Sampling Location	Identification (ID) Number	Matrix	Depth (feet)[1]	Analytical Group	Number of Samples (identify field duplicates)	Sampling SOP Reference
JAX45-DPT16	JAX45-DPT16-60-Date	Water	60-64	VOCs, SVOCs, Low Level PAHs, PCBs, Metals, and TRPH	1	FS 1000, FS 2000, and FS 2200
JAX45-DPT17	JAX45-DPT17-12-Date	Water	12-16	VOCs, SVOCs, Low Level PAHs, PCBs, Metals, and TRPH	1	FS 1000, FS 2000, and FS 2200
JAX45-DPT17	JAX45-DPT17-20-Date	Water	20- 24	VOCs, SVOCs, Low Level PAHs, PCBs, Metals, and TRPH	1	FS 1000, FS 2000, and FS 2200
JAX45-DPT17	JAX45-DPT17-40-Date	Water	40–44	VOCs, SVOCs, Low Level PAHs, PCBs, Metals, and TRPH	1	FS 1000, FS 2000, and FS 2200
JAX45-DPT17	JAX45-DPT17-60-Date	Water	60-64	VOCs, SVOCs, Low Level PAHs, PCBs, Metals, and TRPH	1	FS 1000, FS 2000, and FS 2200
JAX45-DPT18	JAX45-DPT18-12-Date	Water	12-16	VOCs, SVOCs, Low Level PAHs, PCBs, Metals, and TRPH	1	FS 1000, FS 2000, and FS 2200
JAX45-DPT18	JAX45-DPT18-20-Date	Water	20- 24	VOCs, SVOCs, Low Level PAHs, PCBs, Metals, and TRPH	1	FS 1000, FS 2000, and FS 2200

Sampling Location	Identification (ID) Number	Matrix	Depth (feet)[1]	Analytical Group	Number of Samples (identify field duplicates)	Sampling SOP Reference
JAX45-DPT18	JAX45-DPT18-40-Date	Water	40–44	VOCs, SVOCs, Low Level PAHs, PCBs, Metals, and TRPH	1	FS 1000, FS 2000, and FS 2200
JAX45-DPT18	JAX45-DPT18-60-Date	Water	60-64	VOCs, SVOCs, Low Level PAHs, PCBs, Metals, and TRPH	1	FS 1000, FS 2000, and FS 2200
GROUNDWATER SAMPLES MONITORING WELLS						
JAX45-GW-MW01	JAX45-GW-MW01-Depth-Date	Water	TBD[2]	VOCs, SVOCs, Low Level PAHs, PCBs, Metals, and TRPH	1	FS 1000, FS 2000, and FS 2200
JAX45-GW-MW02	JAX45-GW-MW02-Depth-Date	Water	TBD[2]	VOCs, SVOCs, Low Level PAHs, PCBs, Metals, and TRPH	1	FS 1000, FS 2000, and FS 2200
FIELD DUPLICATES DPT						
JAX45-DPT-DUP01	JAX45-DPT-DUP01-Depth-Date	Water	TBD[2]	VOCs, SVOCs, Low Level PAHs, PCBs, Metals, and TRPH	1	FS 1000, FS 2000, and FS 2200
JAX45-DPT-DUP02	JAX45-DPT-DUP02-Depth-Date	Water	TBD[2]	VOCs, SVOCs, Low Level PAHs, PCBs, Metals, and TRPH	1	FS 1000, FS 2000, and FS 2200

Sampling Location	Identification (ID) Number	Matrix	Depth (feet)[1]	Analytical Group	Number of Samples (identify field duplicates)	Sampling SOP Reference
FIELD DUPLICATE MONITORING WELLS						
JAX45-GW-DUP01	JAX45-GW-DUP01-Depth-Date	Water	TBD[2]	VOCs, SVOCs, Low Level PAHs, PCBs, Metals, and TRPH	1	FS 1000, FS 2000, and FS 2200

1 In general, groundwater samples will be collected from the intervals as shown. The actual sampling interval location is subject to change based on the lithology boring data. If any significant clay units (greater than 2-feet thick) are found, then a groundwater sample will be collected from the interval immediately above it. The boring will not be advanced through a significant clay unit.

2 To be determined.

SAP Worksheet #18.2 –Soil Sampling Locations and Methods/SOP Requirements Table

Sampling Location	Identification (ID) Number	Matrix	Depth (feet)	Analytical Group	Number of Samples (identify field duplicates)	Sampling SOP Reference
JAX45-SB05	JAX45-SB05-SB-Date	Soil	0 to 2, Top of water table plus 2 feet	VOCs, SVOCs, Low Level PAHs, PCBs, Metals, and TRPH	1	CT-04, CT-05, SA-6.3, SA-7.1, FS 3000 or Tetra Tech SOP SA 2.5
JAX45-SB06	JAX45-SB06-SB-Date	Soil	0 to 2, Top of water table plus 2 feet	VOCs, SVOCs, Low Level PAHs, PCBs, Metals, and TRPH	1	CT-04, CT-05, SA-6.3, SA-7.1, FS 3000 or Tetra Tech SOP SA 2.5
JAX45-SB07	JAX45-SB07-SB-Date	Soil	0 to 2, Top of water table plus 2 feet	VOCs, SVOCs, Low Level PAHs, PCBs, Metals, and TRPH	1	CT-04, CT-05, SA-6.3, SA-7.1, FS 3000 or Tetra Tech SOP SA 2.5
JAX45-SB08	JAX45-SB08-SB-Date	Soil	0 to 2, Top of water table plus 2 feet	VOCs, SVOCs, Low Level PAHs, PCBs, Metals, and TRPH	1	CT-04, CT-05, SA-6.3, SA-7.1, FS 3000 or Tetra Tech SOP SA 2.5
JAX45-SB09	JAX45-SB09-SB-Date	Soil	0 to 2, Top of water table plus 2 feet	VOCs, SVOCs, Low Level PAHs, PCBs, Metals, and TRPH	1	CT-04, CT-05, SA-6.3, SA-7.1, FS 3000 or Tetra Tech SOP SA 2.5
JAX45-SB10	JAX45-SB10-SB-Date	Soil	0 to 2, Top of water table plus 2 feet	VOCs, SVOCs, Low Level PAHs, PCBs, Metals, and TRPH	1	CT-04, CT-05, SA-6.3, SA-7.1, FS 3000 or Tetra Tech SOP SA 2.5

Sampling Location	Identification (ID) Number	Matrix	Depth (feet)	Analytical Group	Number of Samples (identify field duplicates)	Sampling SOP Reference
JAX45-SB11	JAX45-SB11-SB-Date	Soil	0 to 2, Top of water table plus 2 feet	VOCs, SVOCs, Low Level PAHs, PCBs, Metals, and TRPH	1	CT-04, CT-05, SA-6.3, SA-7.1, FS 3000 or Tetra Tech SOP SA 2.5
JAX45-SB12	JAX45-SB12-SB-Date	Soil	0 to 2, Top of water table plus 2 feet	VOCs, SVOCs, Low Level PAHs, PCBs, Metals, and TRPH	1	CT-04, CT-05, SA-6.3, SA-7.1, FS 3000 or Tetra Tech SOP SA 2.5
JAX45-SB13	JAX45-SB13-SB-Date	Soil	0 to 2, Top of water table plus 2 feet	VOCs, SVOCs, Low Level PAHs, PCBs, Metals, and TRPH	1	CT-04, CT-05, SA-6.3, SA-7.1, FS 3000 or Tetra Tech SOP SA 2.5
JAX45-SB14	JAX45-SB14-SB-Date	Soil	0 to 2, Top of water table plus 2 feet	VOCs, SVOCs, Low Level PAHs, PCBs, Metals, and TRPH	1	CT-04, CT-05, SA-6.3, SA-7.1, FS 3000 or Tetra Tech SOP SA 2.5
FIELD DUPLICATES - SOIL						
JAX45-SB-DUP01	JAX45-DUP01-Date	Soil	TBD	VOCs, SVOCs, Low Level PAHs, PCBs, Metals, and TRPH	1	CT-04, CT-05, SA-6.3, SA-7.1, FS 3000 or Tetra Tech SOP SA 2.5

SAP Worksheet #19 – Analytical SOP Requirements Table
(UFP-QAPP Manual Section 3.1.1)

Matrix	Analytical Group	Analytical and Preparation Method / SOP Reference[1]	Containers (number, size, and type)	Sample Volume (units)	Preservation Requirements	Maximum Holding Time (preparation / analysis)
Soil	VOCs	SW846 5035, 8260B / CA-202, CA-214	Three terra cores sample vials [5 gram (g) size]	5 g (each)	Sodium bisulfate / methanol/ water and freeze to -10 °C	48 hours from sampling to preparation, 14 days to analysis
Groundwater and Aqueous QC Samples	VOCs	SW846 5030, 8260B / CA-202	Three 40 mL glass vials	40 mL	Hydrochloric acid (HCl) to pH<2; Cool to 0 to 6 °C; no headspace	14 days to analysis
Soil	SVOCs (including PAHs by SIM)	SW846 3545A, 3550C, 8270D, 8270D SIM / CA-213, CA-226, CA-512, CA-526	One 8-ounce (oz) wide-mouth glass jar	30 g	Cool to 0 to 6 °C	14 days to extract / 40 days from extraction to analysis
Groundwater and Aqueous QC Samples	SVOCs (including PAHs by SIM)	SW846 3510C, 3520C, 8270D, 8270D SIM / CA-213, CA-226, CS-502	Two 1 Liter (L) amber glass bottles	1 L	Cool to 0 to 6 °C	7 days to extract / 40 days from extraction to analysis
Soil	PCBs	SW846 3540C, 3545A, 3550C, 8082A / CA-329, CA-500, CA-524, CA-537	One 8 oz wide-mouth glass jar	30 g	Cool to 0 to 6 °C	14 days to extract / 40 days from extraction to analysis
Groundwater and Aqueous QC Samples	PCBs	SW846 3510C, 3520C 8082A / CA-329, CA-515	Two 1 L amber glass bottles	1 L	Cool to 0 to 6 °C	7 days to extract / 40 days from extraction to analysis
Soil	Metals (Including Mercury)	SW846 3050B, 6010C, 7471A / CA-605, CA-608, CA-611	One 4 oz wide-mouth glass jar	2 g / 0.3 g for mercury	Cool to 0 to 6 °C	6 months to analysis for all except mercury; mercury is 28 days to analysis.
Groundwater and Aqueous QC Samples	Metals (Including Mercury)	SW846 3010A, 6010C, 7470A / CA-604, CA-608, CA-615	One 1 L High Density Polyethylene bottle	200 mL	Nitric acid to pH <2; Cool to 0 to 6 °C	6 months to analysis for all except mercury; mercury is 28 days to analysis.
Soil	TRPH	FL-PRO[2]/ CA-333	One 4 oz glass jar	30 g	Cool to 0 to 6 °C	7 days until extraction, 40 days to analysis

Matrix	Analytical Group	Analytical and Preparation Method / SOP Reference[1]	Containers (number, size, and type)	Sample Volume (units)	Preservation Requirements	Maximum Holding Time (preparation / analysis)
Groundwater and Aqueous QC Samples	TRPH	FL-PRO[2]/ CA-333	1,000 mL	Two 1 - L glass amber bottles	HCl to pH <2; Cool to 0 to 6 °C	7 days until extraction/40 days to analysis

1 Laboratory SOPs are subject to revision and updates during duration of the project. The laboratory will use the most current revision of the SOP at the time of analysis.

2 FL-PRO – Florida Residual Petroleum Organic Method

SAP Worksheet #20 – Field Quality Control Sample Summary Table
(UFP-QAPP Manual Section 3.1.1)

Matrix	Analytical Group	No. of Sampling Locations[1]	No. of Field Duplicates	No. of MS/MSDs[2]	No. of Field Blanks	No. of VOC Trip Blanks[3]	No. of Equip. Blanks[4]	Total No. of Samples to Lab
Groundwater	VOCs, SVOCs,Low Level PAHs, PCBs, Metals, and TRPH	28	3	2/2	0	2	2	35
Soil	VOCs, SVOCs,Low Level PAHs PCBs, Metals, and TRPH	10	1	1/1	0	NA	1	12

1 If samples will be collected at different depths at the same location, count each discrete sampling depth as a separate sampling location or station.

2 Although the MS/MSD (or MS/sample duplicate for metals) are not typically considered field QC samples and are not included in the "Total No. of Samples to Lab", they are included here because location determination is often established in the field.

3 Trip Blanks will only be analyzed for VOCs and will only be submitted with groundwater samples (one per cooler of aqueous VOC samples).

4 Equipment Rinsate Blanks may not be necessary if the samples are collected using dedicated sampling equipment or if there is no equipment used to collect the samples.

SAP Worksheet #21 – Project Sampling SOP References Table
(UFP-QAPP Manual Section 3.1.2)

Reference Number	Title, Revision Date and/or Number[1]	Originating Organization of Sampling SOP	Equipment Type	Modified for Project Work? (Y/N)	Comments
CT-04	Sample Nomenclature (Revision 2, 03/09/09)	Tetra Tech	NA	N	SOPs are included in Appendix C.
CT-05	Database Records and Quality Assurance (Revision 2, 01/29/01)	Tetra Tech	NA	N	
FS 2200	Groundwater March 31, 2008 (Effective 12/3/08)	FDEP	NA	N	
FS 3000	Soil March 31, 2008 (Effective 12/3/08)	FDEP	NA	N	
FM 1000	Field Planning and Mobilization, December 2008	FDEP	Equipment supply and preparation and assemble field record supplies.	N	
FQ 1000	Field Quality Control Requirements, December 2008	FDEP	NA	N	
FT 1000	Field Testing General (12/3/08)	FDEP	NA	N	
FT 1100	Field pH (12/3/08)	FDEP	Instruments capable of measuring Hydrogen Ion Activity (pH)	N	
FT 1200	Field Specific Conductance (12/3/08)	FDEP	Instruments capable of measuring Specific Conductance (Conductivity)	N	
FT 1300	Field Salinity (12/3/08)	FDEP	Instruments capable of measuring Salinity	N	

Reference Number	Title, Revision Date and/or Number[1]	Originating Organization of Sampling SOP	Equipment Type	Modified for Project Work? (Y/N)	Comments
FT 1500	Field Dissolved Oxygen (12/3/08)	FDEP	Instruments capable of measuring temperature	N	SOPs are included in Appendix C.
FT 1600	Field Turbidity (12/3/08)	FDEP	Instruments capable of measuring dissolved oxygen	N	
SA-1.1	Site Reconnaissance, Revision 7, April 2008	Tetra Tech	Safety equipment, maps, geologic tools, monitoring equipment, marking items, and field notebooks	N	
SA-2.2	Air Monitoring and Sampling	Tetra Tech	Photo Ionization Detector (PID) or Flame Ionization Detector (FID)	N	
SA-2.5	Direct Push Technology (Geoprobe®/Hydropunch™), Revision 3, September 2003	Tetra Tech	Sampling kit, macrocore sampler, probe sampling adapters, roto hammer with bit	N	
SA-6.1	Non-Radiological Sample Handling Revision 3, February 2004	Tetra Tech	Sample Bottle Ware, Packaging Material, Shipping Materials	N	
SA-6.3	Field Documentation Revision 3, 03/09/09)	Tetra Tech	Field Logbook, Field Sample Forms, Boring Logs	N	
SA-7.1	Decontamination of Field Equipment (Revision 6, 01/28/09)	Tetra Tech	Decontamination Equipment (scrub brushes, phosphate free detergent, de-ionized water)	N	

[1] FDEP Field SOPs can be obtained at the following website: http://www.dep.state.fl.us/labs/qa/sops.htm

SAP Worksheet #22 – Field Equipment Calibration, Maintenance, Testing, and Inspection Table
(UFP-QAPP Manual Section 3.1.2.4)

Field Equipment	Activity[1]	Frequency	Acceptance Criterion	Corrective Action	Responsible Person	SOP Reference[2,3]	Comments
Disposable Hand Trowel – Soil Sampling	Inspection	Per Use	NA	Replace	Tetra Tech FOL or designee	FS 3000	None
PID	Visual Inspection, Calibration	Daily, before use	Manufacturer's Guidance	Replace	Tetra Tech FOL or designee	SA-2.2, Manufacturer's Guidance	None
GPS	Positioning	Beginning and end of each day used	Accuracy: sub-meter horizontal dilution of precision (HDOP)< 3, number of satellites must be at least six	Wait for better signal, replace unit, or choose alternate location technique	Tetra Tech FOL or designee	SOP-01	None

1 Activities may include calibration, verification, testing, maintenance, and/or inspection.
2 Specify the appropriate reference letter or number from the Project Sampling SOP References Table (Worksheet #21).
3 FDEP Field SOPs can be obtained at the following website: http://www.dep.state.fl.us/labs/qa/sops.htm

SAP Worksheet #23 – Analytical SOP References Table
(UFP-QAPP Manual Section 3.2.1)

Laboratory SOP Number	Title, Revision Date, and / or Number	Definitive or Screening Data	Matrix and Analytical Group	Instrument	Organization Performing Analysis	Modified for Project Work?[1] (Y/N)
CA-202	Analysis of VOCs Using Purge and Trap Gas Chromatography/Mass Spectrometer (GC/MS): SW-846 Method 8260, 04/10, Revision 11.	Definitive	Soil, Groundwater, and Aqueous QC Samples - VOCs	GC/MS	Katahdin	N
CA-213	Analysis of SVOCs using SW 846 Method 8270 – Modified For Selected Ion Monitoring (SIM), 04/10, Revision 8.	Definitive	Soil, Groundwater, and Aqueous QC Samples - Low Level PAHs	GC/MS	Katahdin	N
CA-214	Closed-System Purge-And-Trap And Extraction For Volatile Organics In Soil And Waste Samples Using SW846 Method 5035, 09/08, Revision 5.	Definitive	Soil - VOCs	TEKMAR, ARCON, ENCON	Katahdin	N
CA-226	Analysis of SVOCs using Capillary Column GC/MS: SW-846 Method 8270D, 08/09, Revision 1.	Definitive	Soil, Groundwater, and Aqueous QC Samples - SVOCs	GC/MS	Katahdin	N
CA-329	Analysis of PCBs as Total Aroclors By Gas Chromatography/Electron Capture Detector (GC/ECD): SW-846 Method 8082, 04/10, Revision 11.	Definitive	Soil, Groundwater, and Aqueous QC Samples - PCBs	GC/ECD	Katahdin	Y – final extract volume of 2 mL
CA-333	Determination of Petroleum Range Organics using Florida Department of Environmental Protection Method FL-PRO, 09/08, Revision 4.	Definitive	Soil, Groundwater, and Aqueous QC Samples - TRPH	Gas Chromatography/ Flame Ionization Detector (GC/FID)	Katahdin	N

Laboratory SOP Number	Title, Revision Date, and / or Number	Definitive or Screening Data	Matrix and Analytical Group	Instrument	Organization Performing Analysis	Modified for Project Work?[1] (Y/N)
CA-500	Preparation of Sediment/Soil Samples by Sonication using Method 3550 for Subsequent Pesticides/PCBs Analysis, 02/09, Revision 6.	Definitive	Soil - Pesticides and PCBs Extractions	Sonicator	Katahdin	N
CA-502	Preparation of Aqueous Samples for Extractable Semivolatile Analysis, 10/09, Revision 6.	Definitive	Groundwater and Aqueous QC Samples - SVOCs/ PAHs Extractions	Separatory Funnel, Continuous liquid to liquid extraction (CLLE)	Katahdin	N
CA-512	Preparation of Sediment/Soil Samples by Sonication using Method 3550 for Subsequent Extractable Semivolatiles Analysis, 02/09, Revision 7.	Definitive	Soil - SVOCs/PAHs Extractions	Sonicator	Katahdin	N
CA-515	Preparation of Aqueous Samples for Pesticides/PCBs Analysis, 10/09, Revision 6.	Definitive	Groundwater and Aqueous QC Samples - PCBs Extractions	Separatory Funnel, CLLE	Katahdin	N
CA-524	Preparation of Sediment/Soil Samples by Soxhlet Extraction using Method 3540 for Pesticide/PCB Analysis, 08/09, Revision 6.	Definitive	Soil - PCBs Extractions	Soxhlet	Katahdin	N
CA-526	Preparation of Sediment/Soil Samples by Soxhlet Extraction using Method 3540 For Subsequent Extractable Semivolatile Analysis, 08/09, Revision 6.	Definitive	Soil - SVOCs/PAHs Extractions	Soxhlet	Katahdin	N
CA-537	Preparation of Sediment/Soil and Tissue Samples by Accelerated Solvent Extraction using Method 3545 for Subsequent Extractable Pesticide and PCB Analysis, 08/09, Revision 2.	Definitive	Soil - PCBs Extractions	Accelerate Solvent Extraction	Katahdin	N

Laboratory SOP Number	Title, Revision Date, and / or Number	Definitive or Screening Data	Matrix and Analytical Group	Instrument	Organization Performing Analysis	Modified for Project Work?[1] (Y/N)
CA-604	Acid Digestion of Aqueous Samples using USEPA Method 3010 for Inductively Coupled Plasma (ICP) Analysis of Total or Dissolved Metals, 04/10, Revision 5.	Definitive	Groundwater and Aqueous QC Samples - Metals Digestion	Block Digester	Katahdin	N
CA-605	Acid Digestion of Solid Samples using USEPA Method 3050 For Metals Analysis by ICP-Atomic Emission Spectroscopy (AES) and Graphite Furnace Atomic Absorption, 08/09, Revision 4.	Definitive	Soil - Metals Digestion	Block Digester	Katahdin	N
CA-608	Trace Metals Analysis by ICP-AES using USEPA Method 6010, 06/10, Revision 11.	Definitive	Soil, Groundwater, and Aqueous QC Samples - Metals	ICP-AES	Katahdin	N
CA-611	Digestion and Analysis of Solid Samples for Mercury By USEPA Method 7471, 04/10, Revision 7.	Definitive	Soil - Mercury	Cold Vapor Atomic Absorption (CVAA)	Katahdin	N
CA-615	Digestion and Analysis of Aqueous Samples for Mercury By USEPA Method 7470, 04/10, Revision 5.	Definitive	Groundwater and Aqueous QC Samples - Mercury	CVAA	Katahdin	N
SD-902	Sample Receipt and Internal Control, 08/09, Revision 8.	Definitive	Sample Receiving	NA	Katahdin	N
SD-903	Sample Disposal, 05/09, Revision 4.	Definitive	Sample Receiving	NA	Katahdin	N

SAP Worksheet #24 – Analytical Instrument Calibration Table
(UFP-QAPP Manual Section 3.2.2)

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action	Person Responsible for Corrective Action	SOP Reference[1]
GC/MS VOCs	Initial Calibration (ICAL) – A minimum 5-point calibration is required	Calibrate the instrument when it is received, after a major change (source cleaning, new column, change in GC run parameters), or if the daily calibration fails.	The average Response Factors (RFs) for System Performance Check Compounds (SPCCs) are 1,1,2,2-tetrachloroethane and chlorobenzene must be ≥0.30 and chloromethane, 1,1-dichloroethane, and bromoform must be ≥0.10. The Percent Relative Standard Deviations (%RSDs) for RFs of Calibration Check Compounds (CCCs) must be ≤ 30%, and the %RSDs must be ≤ 15% for all target analytes. If not met: Option 1) Linear least squares regression: Linear Regression Correlation Coefficient (r) must be ≥ 0.995; or Option 2) Non-linear regression: coefficient of determination (r^2) must be ≥ 0.990 (6 points are required for second order).	Repeat calibration if criterion is not met.	Analyst, Supervisor	CA-202

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action	Person Responsible for Corrective Action	SOP Reference[1]
GC/MS VOCs (continued)	Retention Time (RT) Window Position Establishment	Once per ICAL for each analyte and surrogate.	Position shall be set using the midpoint standard of the ICAL curve when ICAL is performed. On days when ICAL is not performed, the initial continuing calibration verification is used.	NA.	Analyst, Supervisor	CA-202
	Evaluation of Relative Retention Times (RRTs)	With each sample.	RRT of each target analyte must be within ± 0.06 RRT units.	Correct problem, then rerun ICAL.	Analyst, Supervisor	
	Initial Calibration Verification (ICV) – Second Source	Once after each ICAL prior to sample analysis.	Percent Recovery (%R) must be within 80-120% for all target analytes.	Correct problem and verify ICV. Reanalyze ICV and/or ICAL as appropriate.	Analyst, Supervisor	
	Continuing Calibration Verification (CCV)	Analyze a standard at the beginning of each 12-hour shift after a bromofluorobenzene (BFB) tune.	Percent Difference or Percent Drift (%D) must be ≤ 20% for all target analytes and surrogates. RFs for SPCCs must be ≥0.10 and ≥0.30 (compounds as listed above in ICAL block).	Correct problem, then rerun calibration verification. If that fails, then repeat ICAL. Reanalyze all samples since last acceptable CCV.	Analyst, Supervisor	
	BFB Tune	Prior to ICAL and at the beginning of each 12-hour analytical sequence.	Criteria listed in Table 4 of Katahdin SOP CA-202. No samples may be analyzed without a valid tune.	Retune and/or clean source.	Analyst, Supervisor	

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action	Person Responsible for Corrective Action	SOP Reference[1]
GC/MS SVOCs (including PAHs by SIM)	Breakdown Check (DDT only)	At the beginning of each 12-hour analytical sequence.	The degradation must be ≤ 20% for DDT to verify inertness of the injection port.	Correct the problem then repeat breakdown check. No samples shall be run until degradation is ≤20% for DDT.	Analyst, Supervisor	CA-213, CA-226
	ICAL – A minimum 5-point calibration is required	Calibrate the instrument when it is received, after a major change (source cleaning, new column, change in GC run parameters), or if the daily calibration fails.	Average RF SPCCs must be ≥0.050 (≥0.010 for SIM); %RSD for RFs for CCCs must be ≤30%; and the %RSD must be ≤ 15% for all other compounds. If not met: Option 1) r must be ≥ 0.995, or Option 2) r^2 must be ≥ 0.99 (minimum of 6 points required for second order). For low-level SVOCs and PAHs, the %RSD must be ≤ 20%, or meet one of the above options.	Recalibrate and/or perform the necessary equipment maintenance. Check the calibration standards.	Analyst, Supervisor	
	ICV – Second Source	Once after each ICAL prior to sample analysis	%R must be within 80-120% for all target analytes.	Correct problem and verify second source standard. Reanalyze ICV and/or ICAL as appropriate.	Analyst, Supervisor	

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action	Person Responsible for Corrective Action	SOP Reference[1]
GC/MS SVOCs (including PAHs by SIM)	RT Window Position Establishment	Once per ICAL for each analyte and surrogate.	Position shall be set using the midpoint standard of the ICAL curve when ICAL is performed. On days when ICAL is not performed, the initial CCV is used.	NA.	Analyst, Supervisor	CA-213, CA-226
	Evaluation of RRTs	With each sample.	RRT of each target analyte must be within ± 0.06 RRT units.	Correct problem, then rerun ICAL.	Analyst, Supervisor	
	CCV	Analyze a standard at the beginning of each 12-hour shift after a decafluorotriphenyl-phosphine (DFTPP) tune.	%D must be ≤ 20% for all target analytes and surrogates. SPCCs RFs must be >0.050 (≥0.010 for SIM).	Recalibrate and/or perform the necessary equipment maintenance. Check the calibration standards. Reanalyze the affected data.	Analyst, Supervisor	
	DFTPP Tune	Prior to initial calibration and at the beginning of each 12-hour analytical sequence.	Criteria listed in Section 7.4 of Katahdin SOP CA-213 and Section 7.4 of CA-226. No samples may be analyzed without a valid tune.	Retune and/or clean source.	Analyst, Supervisor	

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action	Person Responsible for Corrective Action	SOP Reference[1]
GC/ECD PCBs	ICAL – A minimum 5- point calibration curve is run for Aroclor 1016 and 1260 and a single-point reference for all other Aroclors. If an Aroclor other than 1016/1260 is identified in any sample by peak pattern, then the sample is re-analyzed with a full calibration curve for that Aroclor	Instrument receipt, major instrument change, when CCV does not meet criteria.	6-point calibration of Aroclors 1016/1260, 1242, 1248, and 1254 – One of the options below: Option 1: RSD for each analyte must be ≤ 20%; Option 2: r must be ≥ 0.995; Option 3: r^2 must be ≥ 0.99 (6 points shall be used for second order). Midpoint calibration of Aroclors 1221 and 1232; if these target analytes are detected, a 6-point calibration is performed and the samples are reanalyzed.	Repeat ICAL and/or perform necessary equipment maintenance. Check calibration standards. Reanalyze affected data.	Analyst, Supervisor	CA-329
	ICV – Second Source	Once after each ICAL and prior to sample analysis.	%R must be within 80-120% for all target analytes.	Identify source of problem, correct, repeat calibration, rerun samples.	Analyst, Supervisor	
	CCV	Once after each ICAL and at the beginning and end of each run sequence and every 10 samples.	%D of all target analytes must be ≤ 20%.	Identify source of problem, correct, repeat calibration, rerun samples.	Analyst, Supervisor	

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action	Person Responsible for Corrective Action	SOP Reference[1]
ICP-AES Metals (Except Mercury)	ICAL – One point calibration for each element	Daily prior to sample analysis, and if continuing QC fails.	None; only one high standard and a calibration blank must be analyzed. If more than one calibration standard is used, r must be ≥ 0.995.	Recalibrate and/or perform necessary equipment maintenance. Check calibration standards.	Analyst, Supervisor	CA-608
	ICV – Second Source	Once after each ICAL and prior to sample analysis.	%R must be within 90-110% of true value.	Do not use results for failing elements, unless ICV >110% and sample result < reporting limit.	Analyst, Supervisor	
	CCV	At the beginning and end of each run sequence and every 10 samples.	%R must be within 90-110% of true value.	Check problem, recalibrate and reanalyze any samples not bracketed by passing CCVs.	Analyst, Supervisor	
	Initial Calibration Blank (ICB)	Before beginning a sample sequence.	No analyte detected > LOD.	Correct the problem, then re-prepare and reanalyze.	Analyst, Supervisor	
	Continuing Calibration Blank (CCB)	After the initial CCV, after every 10 samples, and at the end of the sequence	No analyte detected > LOD.	Correct the problem, then re-prepare and reanalyze calibration blank and all affected samples.	Analyst, Supervisor	

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action	Person Responsible for Corrective Action	SOP Reference[1]
ICP-AES Metals (Except Mercury)	Low-Level Calibration Check Standard (if using 1-point ICAL)	At beginning and end of run.	%R must be within 80%-120% of true value.	Do not use results for failing elements, unless LOQ recovery > upper limit and sample result < LOQ/reporting limit.	Analyst, Supervisor	CA-608
	Interference Check Standards (ICS) – ICS A and ICSA B)	Daily, before sample injections.	ICS A recoveries must be less than the absolute value of the LOD and ICSA B %Rs must be within 80-120% of the true value.	Correct the problem, then re-prepare checks and reanalyze all affected samples.	Analyst, Supervisor	
CVAA Mercury	ICAL – A 6-point calibration curve is analyzed	Daily prior to sample analysis, and if continuing QC fails.	The RSD for RFs must be ≤ 20% or r must be ≥ 0.995.	Recalibrate and/or perform necessary equipment maintenance. Check calibration standards	Analyst, Supervisor	CA-611, CA-615
	ICB	Before beginning a sample sequence.	No mercury detected > LOD.	Correct problem, reprepare, and reanalyze.	Analyst, Supervisor	
	ICV – Second Source	Once after each ICAL and prior to sample analysis	%R must be within 90-110% of the true value.	Correct problem and repeat calibration.	Analyst, Supervisor	
	CCB	After each CCV, after every 10 samples, and at the end of the sequence	No mercury detected > LOD.	Investigate source of contamination, rerun any samples not bracketed by passing blanks	Analyst, Supervisor	

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action	Person Responsible for Corrective Action	SOP Reference[1]
CVAA Mercury	CCV	CCV-at beginning and end of each run sequence and every 10 samples.	%R must be within 80-120% of the true value.	Check problem, recalibrate and reanalyze any samples not bracketed by passing CCVs.	Analyst, Supervisor	CA-611, CA-615
GC/FID TRPH	ICAL – A minimum of a 5-point calibration is prepared for all target analytes	Upon instrument receipt, major instrument change, or when the CCV does not meet criteria.	The RSD for RFs for each target analyte must be ≤ 20%, or r must be ≥ 0.995.	Correct problem then repeat ICAL. No samples may be run until ICAL has passed.	Analyst, Supervisor	CA-333
	ICV – Second Source	Following ICAL, prior to the analysis of samples.	The %R must be within 80-120% of true value.	Correct problem and verify ICV. If that fails, correct problem and repeat ICAL. No samples may be run until ICV has been verified.	Analyst, Supervisor	
	CCV	At the beginning of a sequence and after every 12 hours or 10 samples (whichever comes first), then at the end of the sequence.	The %R must be within 75-125% of true value.	Correct problem and rerun CCV. If that fails, repeat ICAL and reanalyze all samples analyzed since the last successful CCV. If the CCV fails high, report samples that are less than the LOQ.	Analyst, Supervisor	

[1] Laboratory SOPs are subject to revision and updates during duration of the project, the laboratory will use the most current revision of the SOP at the time of analysis.

SAP Worksheet #25 – Analytical Instrument and Equipment Maintenance, Testing, and Inspection Table
(UFP-QAPP Manual Section 3.2.3)

Instrument / Equipment	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Responsible Person	SOP Reference
GC/MS	Check pressure, gas supply and vacuum daily. Bake out trap and column, manual tune if BFB not in criteria, change septa as needed, cut column as needed, change trap as needed, clean MS source as needed. Other maintenance specified in Laboratory Equipment Maintenance SOP.	VOCs	Ion source, injector liner, column, column flow, purge lines, purge flow, trap.	Prior to ICAL and/or as necessary.	Acceptable ICAL or CCV.	Correct the problem and repeat ICAL or CCV.	Analyst, Supervisor	CA-202
GC/MS	Check pressure, gas supply, and vacuum daily. Bake out column, manual tune if DFTPP not in criteria, change septa as needed, cut column as needed, clean MS source as needed. Other maintenance specified in Laboratory Equipment Maintenance SOP.	SVOCs (including Low Level PAHs by SIM)	Ion source, injector liner, column, column flow.	Prior to ICAL and/or as necessary.	Acceptable ICAL or CCV.	Correct the problem and repeat ICAL or CCV.	Analyst, Supervisor	CA-213, CA-226
GC/ECD	Check pressure and gas supply daily. Change septa and/or liner as needed, replace or cut column as needed. Other maintenance specified in Laboratory Equipment Maintenance SOP.	PCBs	Injector liner, septa, column, column flow.	Prior to ICAL and/or as necessary.	Acceptable ICAL or CCV.	Correct the problem and repeat ICAL or CCV.	Analyst, Supervisor	CA-329

Instrument / Equipment	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Responsible Person	SOP Reference
ICP-AES	Clean sample path, check pump tubing, argon level, vacuum and waste container daily. Clean source as needed. Other maintenance specified in Laboratory Equipment Maintenance SOP.	Metals (except mercury)	Pump, pump tubing, vacuum source, waste container.	Prior to ICAL and as necessary.	Acceptable ICAL or CCV.	Correct the problem and repeat ICAL or CCV.	Analyst, Supervisor	CA-608
CVAA	Replace peristaltic pump tubing, replace mercury lamp, replace drying tube, clean optical cell and/or clean liquid/gas separator as needed. Other maintenance specified in Laboratory Equipment Maintenance SOP.	Mercury	Tubing, sample probe, optical cell, waste container.	Prior to ICAL and as necessary.	Acceptable ICAL or CCV.	Correct the problem and repeat ICAL or CCV.	Analyst, Supervisor	CA-611, CA-615
GC-FID	Check pressure and gas supply daily. Change septa and/or GC injector glass liner as needed. Replace or cut GC column as needed. Other maintenance specified in Laboratory Equipment Maintenance SOP.	TRPH	Injector liner, septa, column, column flow.	Prior to ICAL and as necessary.	Acceptable ICAL or CCV.	Correct the problem and repeat ICAL or CCV.	Analyst, Supervisor	CA-333

SAP Worksheet #26 – Sample Handling System
(UFP-QAPP Manual Appendix A)

SAMPLE COLLECTION, PACKAGING, AND SHIPMENT
Sample Collection (Personnel/Organization): Tetra Tech FOL or designee / Tetra Tech
Sample Packaging (Personnel/Organization): Tetra Tech FOL or designee / Tetra Tech
Coordination of Shipment (Personnel/Organization): Tetra Tech FOL or designee / Tetra Tech
Type of Shipment/Carrier: Federal Express
SAMPLE RECEIPT AND ANALYSIS
Sample Receipt (Personnel/Organization): Sample Custodians / Katahdin
Sample Custody and Storage (Personnel/Organization): Sample Custodians/ Katahdin
Sample Preparation (Personnel/Organization): Extraction Laboratory, Metals Preparation Laboratory / Katahdin
Sample Determinative Analysis (Personnel/Organization): Gas Chromatography Laboratory, GC/MS Laboratory, Metals Laboratory / Katahdin
SAMPLE ARCHIVING
Field Sample Storage (Number of days from sample collection): 60 days from receipt
Sample Extract/Digestate Storage (No. of days from extraction/digestion): 3 months from sample digestion/extraction
Biological Sample Storage (Number of days from sample collection): N/A
SAMPLE DISPOSAL
Personnel/Organization: Sample Custodians / Katahdin

SAP Worksheet #27 – Sample Custody Requirements Table
(UFP-QAPP Manual Section 3.3.3)

27.1 SAMPLE NOMENCLATURE, SAMPLE COLLECTION DOCUMENTATION, HANDLING, AND TRACKING PROCEDURES

The following sections outline the procedures that will be used to document project activities and sample collection, handling, tracking, and custody procedures during the investigation. All forms must be filled in as completely as possible.

27.1.1 Sample Nomenclature

Refer to Worksheet #18 for how the samples will be labeled. Also, refer to Worksheet #20 for how the field QA/QC samples will be labeled.

Sample nomenclature will be conducted in general accordance with the procedures outlined in Tetra Tech SOP CT-04 (Sample Nomenclature). Sample nomenclature put forth for this field event has been selected based on historical usage. The sample nomenclature for each tracking number includes the site being investigated, sample media identifier, and sample location number. The standard sample matrix and type codes used for this field event are as follows: Duplicate samples will be submitted to the laboratory as blind duplicates. The QA/QC type codes used for this field event are as follows: TB for Trip Blanks and RB for rinsate blanks. Field QC blanks will be labeled sequentially followed by the date (i.e., TB-20101213, FB-20101214, etc.). Samples to be used for MS and MSDs will be labeled MS/MSD on the container label and noted on the chain-of-custody, as required in the laboratory QA Plan; however, "MS/MSD" will not be part of the unique sample identifier in order to maintain consistency with the project database. Additional information regarding protocol for sample labeling is contained in Tetra Tech SOP SA-6.3 (see Appendix C).

27.1.2 Sample Collection Documentation

Documentation of field observations will be recorded in a field logbook and/or field log sheets including sample collection logs, boring logs, VOC screening logs, and monitoring well construction logs. Field logbooks utilized on this project will consist of a bound, water-resistant logbook. All pages of the logbook will be numbered sequentially and observations will be recorded with indelible ink.

Field sample log sheets will be used to document sample collection details and other observations and activities will be recorded in the field logbook. Instrument calibration logs will be used to record the daily instrument calibration. Example field forms are included in Appendix C.

For sampling and field activities, the following types of information will be recorded in the field logbook as appropriate:

- Site name and location
- Date and time of logbook entries
- Personnel and their affiliations
- Weather conditions
- Activities involved with the sampling
- Subcontractor activity summary
- Site observations including site entry and exit times
- Site sketches made on site
- Visitor names, affiliations, arrival and departure times
- Health and safety issues, including PPE

27.1.3 Sample Handling and Tracking System

Following sample collection into the appropriate bottle ware, all samples will be immediately placed on ice in a cooler. The glass sample containers will be enclosed in bubble-wrap in order to protect the bottle ware during shipment. The cooler will be secured using strapping tape along with a signed custody seal. Sample coolers will be delivered to a local courier location for priority overnight delivery to the selected laboratory for analysis. Samples will be preserved as appropriate based on the analytical method. The laboratories will provide pre-preserved sample containers for sample collection. Samples will be maintained at 0 to 6 °C until delivery to the laboratory. Proper custody procedures will be followed throughout all phases of sample collection and handling.

After collection, each sample will be maintained in the sampler's custody until formally transferred to another party (e.g., Federal Express). For all samples collected, chain-of-custody forms will document the date and time of sample collection, the sampler's name, and the names of all others who subsequently held custody of the sample. Specifications for chemical analyses will also be documented on the chain-of-custody form. Tetra Tech SOP SA-6.3 (Field Documentation) provides further details on the chain-of-custody procedure, which is provided in Appendix C.

These subsections outline the procedures that will be used by field and laboratory personnel to document project activities and sample collection procedures during this RI. All forms must be filled in as completely as possible.

Sample handling requirements are described in Worksheet #26. Tetra Tech personnel will collect the samples. The samplers will take care not to contaminate samples through improper handling. Samples will be sealed in appropriate containers, packaged by Tetra Tech personnel and placed into sealed coolers under chain-of-custody in accordance with the applicable SOP (See Worksheet #21). Samples to be analyzed for VOCs will be accompanied by a VOC trip blank. All coolers will contain a temperature blank. Samples will be transferred under chain-of-custody to a courier as described below. Once received by the laboratory, receipt will be documented on the chain-of-custody form and the samples will be checked in. The samples will remain under chain-of-custody throughout the analysis period to ensure their integrity is preserved. Details are provided below.

Samples to be delivered to the laboratory(s) will be made by a public courier (i.e., Federal Express). After samples have been collected, they will be sent to the laboratory(s) within 24 hours. Under no circumstances will sample holding times be exceeded.

27.2 FIELD SAMPLE CUSTODY PROCEDURES

Chain-of-custody protocols will be used throughout sample handling to establish the evidentiary integrity of sample containers. These protocols will be used to demonstrate that the samples were handled and transferred in a manner that would eliminate possible tampering. Samples for the laboratory will be packaged and shipped in accordance with Tetra Tech SOP SA-6.1 (see Appendix C).

A sample is under custody if:

- The sample is in the physical possession of an authorized person.
- The sample is in view of an authorized person after being in his/her possession.
- The sample is placed in a secure area by an authorized person after being in his/her possession.
- The sample is in a secure area, restricted to authorized personnel only.

Custody documentation is designed to provide documentation of preparation, handling, storage, and shipping of all samples collected. A multi-part form is used with each page of the form signed and dated by the recipient of a sample or portion of sample. The person releasing the sample and the person receiving the sample each will retain a copy of the form each time a sample transfer occurs.

Integrity of the samples collected during the RI will be the responsibility of identified persons from the time the samples are collected until the samples, or their derived data, are incorporated into the final report.

The Tetra Tech FOL is responsible for the care and custody of the samples collected until they are delivered to the laboratory or are entrusted to a carrier. When transferring samples, the individuals relinquishing and receiving them will sign, date, and note the time on the chain-of-custody form. This record documents the sample custody transfer from the sampler to the laboratory, often through another person or agency (common carrier). Upon arrival at the laboratory, internal sample custody procedures will be followed as defined in the Laboratory SOPs included in Appendix D.

27.3 LABORATORY CHAIN OF CUSTODY – KATAHDIN

Laboratory sample custody procedures (receipt of samples, archiving, and disposal) will be used according to Katahdin SOPs (see Appendix D). Coolers are received and checked for proper temperature and preservation. A sample cooler receipt form will be filled out to note conditions and any discrepancies. The chain-of-custody will be checked against the sample containers for correctness. Samples will be logged into the Laboratory Information Management System and given a unique log number that can be tracked through processing. The Katahdin Laboratory PM will notify the Tetra Tech FOL of any problems on the same day that the issue is identified.

SAP Worksheet #28 – Laboratory QC Samples Table
(UFP-QAPP Manual Section 3.4)

Matrix	**Soil, Groundwater, and Aqueous QC Samples**
Analytical Group	**VOCs**
Analytical Method/SOP Reference	**SW-846 8260B/ CA-202**

QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	DQI	Measurement Performance Criteria
Method Blank	One is performed for each batch of up to 20 samples.	No target compound concentrations may be > ½ the LOQ, except common laboratory contaminants methylene chloride, acetone, and 2-butanone may be present, but must be < LOQ.	If blank results are above ½ LOQ (or > LOQ for common contaminants), sample results which are < LOQ or > 10X the blank contamination concentration may be reported without corrective action. Otherwise, re-analyze all associated samples.	Analyst, Supervisor, Data Validator	Contamination/ Bias	Same as QC Acceptance Limits.
Laboratory Control Sample (LCS)	One is performed for each batch of up to 20 samples.	%Rs must meet the limits provided in the Katahdin QC Limits table provided in Appendix D.	Correct problem, then reprepare and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available. Contact Client if samples cannot be reanalyzed within hold time.	Analyst, Supervisor, Data Validator	Accuracy/ Bias	Same as QC Acceptance Limits.

Matrix	**Soil, Groundwater, and Aqueous QC Samples**
Analytical Group	**VOCs**
Analytical Method/SOP Reference	**SW-846 8260B/ CA-202**

QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	DQI	Measurement Performance Criteria
MS/MSD	One set is performed for each batch of up to 20 samples.	%Rs should meet the limits provided in the Katahdin QC Limits table provided in Appendix D. The RPD between MS and MSD should be ≤ 30%.	Failure to meet the control limits shall be discussed in the case narrative. If both the LCS and MS/MSD are unacceptable, all associated samples must be re-analyzed.	Analyst, Supervisor, Data Validator	Accuracy / Bias Precision	Same as QC Acceptance Limits.
Surrogates	4 per sample: Dibromofluoromethane 1,2-dichloroethane-d_4 Toluene-d_8 BFB	%Rs must meet the limits provided in the Katahdin QC Limits table provided in Appendix D.	Re-analyze affected samples if volume is available.	Analyst, Supervisor, Data Validator	Accuracy / Bias	Same as QC Acceptance Limits.
Internal Standards (IS)	4 per sample- Pentafluorobenzene 1,4-Difluorobenzene Chlorobenzene-d_5 1,4-Dichlorobenzene-d_4	RTs must be within ± 30 seconds and the response areas must be within -50% to +100% of the last ICAL midpoint standard for each IS.	Re-analyze affected samples if volume is available.	Analyst, Supervisor, Data Validator	Accuracy / Bias	Same as QC Acceptance Limits.
Results between the MDL and LOQ	NA	Apply "J" qualifier to results detected between MDL and LOQ.	None.	Analyst, Supervisor, Data Validator	Precision	Same as QC Acceptance Limits.

Matrix	**Soil, Groundwater, and Aqueous QC Samples**
Analytical Group	**SVOCs (including PAHs by SIM)**
Analytical Method / SOP Reference	**SW-846 8270D, 8270D SIM/ CA-213, CA-226**

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	DQI	Measurement Performance Criteria
Method Blank	One is performed for each batch of up to 20 samples of the same matrix.	No target analytes > ½ the LOQ.	If blank results are above ½ LOQ, sample results which are < LOQ or > 10X the blank contamination concentration may be reported without corrective action. Otherwise, re-extract all associated samples.	Analyst, Supervisor, Data Validator	Contamination / Bias	Same as QC Acceptance Limits.
LCS	One is performed for each batch of up to 20 samples of the same matrix.	%Rs must meet the limits provided in the Katahdin QC Limits table provided in Appendix D.	Correct problem, then re-prepare and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available Contact Client if samples cannot be reanalyzed within hold time.	Analyst, Supervisor, Data Validator	Accuracy / Bias	Same as QC Acceptance Limits.
MS/MSD	One set is performed for each batch of up to 20 samples of the same matrix.	%Rs should meet the limits provided in the Katahdin QC Limits table provided in Appendix D. The RPD between MS and MSD should be ≤ 30%.	Failure to meet the control limits shall be discussed in the case narrative. If both the LCS and MS/MSD are unacceptable, all associated samples must be re-analyzed.	Analyst, Supervisor, Data Validator	Accuracy / Bias Precision	Same as QC Acceptance Limits.

Matrix	**Soil, Groundwater, and Aqueous QC Samples**
Analytical Group	**SVOCs (including PAHs by SIM)**
Analytical Method / SOP Reference	**SW-846 8270D, 8270D SIM/ CA-213, CA-226**

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	DQI	Measurement Performance Criteria
Surrogates	6 per sample: 2-Fluorophenol Phenol-d_6 Nitrobenzene-d_5 2-Fluorobiphenyl 2,4,6-Tribromophenol Terphenyl-d_{14} For low-level PAHs, 3 surrogates per sample: 2-Methylnaphthalene-d_{10} Fluorene-d_{10} Pyrene-d_{10}	%Rs must meet the limits provided in the Katahdin QC Limits table provided in Appendix D.	Field samples having one or more surrogate recoveries above the control limits will not require corrective action if no associated target analytes (acid analytes with acid surrogates, etc.) are detected above the LOQ. Otherwise, affected samples must be reanalyzed.	Analyst, Supervisor, Data Validator	Accuracy / Bias	Same as QC Acceptance Limits.
IS	6 per sample: 1,4-Dichlorobenzene-d_4 Naphthalene-d_8 Acenaphthene-d_{10} Phenanthrene-d_{10} Chrysene-d_{12} Perylene-d_{12}	RTs for ISs must be within ±30 seconds and the response areas must be within -50% to +100% of the last ICAL midpoint standard for each IS.	Re-analyze affected samples if volume is available.	Analyst, Supervisor, Data Validator	Accuracy / Bias	Same as QC Acceptance Limits.
Results between the MDL and LOQ	NA	Apply "J" qualifier to results detected between MDL and LOQ.	None.	Analyst, Supervisor, Data Validator	Precision	Same as QC Acceptance Limits.

Note that limits are updated periodically and may change from the issuance of the final SAP to the time data validation is performed. The limits used for validation will be the limits that are current at the time of analysis.

Matrix	**Soil, Groundwater, and Aqueous QC Samples**
Analytical Group	**PCBs**
Analytical Method / SOP Reference	**SW-846 8082A/ CA-329**

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	DQI	Measurement Performance Criteria
Method Blank	One is performed for each batch of up to 20 samples of the same matrix.	No target compound concentrations may be > ½ the LOQ.	If blank results are above ½ LOQ, sample results which are < LOQ or > 10X the blank contamination concentration may be reported without CA. Otherwise, re-extract all associated samples.	Analyst, Supervisor, Data Validator	Contamination / Bias	Same as QC Acceptance Limits.
LCS	One is performed for each batch of up to 20 samples of the same matrix.	%Rs must meet the limits provided in the Katahdin QC Limits table provided in Appendix D.	Correct problem, then re-prepare and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available Contact Client if samples cannot be re-prepared within hold time.	Analyst, Supervisor, Data Validator	Accuracy/ Bias	Same as QC Acceptance Limits.
MS/MSD	One set is performed for each batch of up to 20 samples of the same matrix.	%Rs should meet the limits provided in the Katahdin QC Limits table provided in Appendix D. The RPD between MS and MSD should be ≤ 30%.	Failure to meet the control limits shall be discussed in the case narrative. If both the LCS and MS/MSD are unacceptable, all associated samples must be re-analyzed.	Analyst, Supervisor, Data Validator	Accuracy/ Bias Precision	Same as QC Acceptance Limits.

Matrix	**Soil, Groundwater, and Aqueous QC Samples**
Analytical Group	**PCBs**
Analytical Method / SOP Reference	**SW-846 8082A/ CA-329**

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	DQI	Measurement Performance Criteria
Surrogates	2 per sample: Decachlorobiphenyl Tetrachloro-m-xylene	%Rs must meet the limits provided in the Katahdin QC Limits table provided in Appendix D.	For QC and field samples, correct problem then re-prepare and reanalyze all failed samples for failed surrogates in the associated preparatory batch, if sufficient sample material is available. If obvious chromatographic interference with surrogate is present, reanalysis may not be necessary. Contact Client if samples cannot be re-prepared within hold time.	Analyst, Supervisor, Data Validator	Accuracy/ Bias	Same as QC Acceptance Limits.
Second Column Confirmation	All positive results must be confirmed.	Results between primary and second column must be RPD ≤ 40%. The higher of the two results will be reported unless matrix interference is apparent.	None. Apply qualifier if RPD >40% and discuss in the case narrative.	Analyst, Supervisor, Data Validator	Precision	Same as QC Acceptance Limits.
Results between the MDL and LOQ	NA	Apply "J" qualifier to results detected between MDL and LOQ.	None.	Analyst, Supervisor, Data Validator	Precision	Same as QC Acceptance Limits.

Note that limits are updated periodically and may change from the issuance of the final SAP to the time data validation is performed. The limits used for validation will be the limits that are current at the time of analysis.

Matrix	**Soil, Groundwater, and Aqueous QC Samples**
Analytical Group	**Metals (Including Mercury)**
Analytical Method/SOP Reference	**SW-846 6010C, 7470A, 7471A/ CA-608, CA-611, CA-615**

QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method Blank	One is performed for each batch of up to 20 samples of the same matrix.	No target analytes > the LOQ.	If blank results are above LOQ, sample results that are < LOQ or > 10X the blank contamination concentration may be reported without corrective action. Otherwise, re-extract all associated samples.	Analyst, Supervisor, Data Validator	Contamination / Bias	Same as QC Acceptance Limits.
LCS	One is performed for each batch of up to 20 samples of the same matrix.	%R must be within 80-120% of the true value.	Failure of any element will necessitate a re-digestion/re-analysis of all associated samples for that element.	Analyst, Supervisor, Data Validator	Accuracy/ Bias	Same as QC Acceptance Limits.
Sample Duplicate	One sample duplicate is performed for each batch of 20 samples of the same matrix.	The RPD should be ≤20%.	Failure to meet the control limit shall be discussed in the case narrative, and elements will be flagged accordingly.	Analyst, Supervisor, Data Validator	Precision	Same as QC Acceptance Limits.
MS	One MS is performed for each batch of up to 20 samples of the same matrix.	%Rs should be within 80-120%, if sample < 4x spike added.	Failure to meet the control limits shall be discussed in the case narrative, and elements will be flagged accordingly.	Analyst, Supervisor, Data Validator	Accuracy/ Bias	Same as QC Acceptance Limits.

Matrix	**Soil, Groundwater, and Aqueous QC Samples**
Analytical Group	**Metals (Including Mercury)**
Analytical Method/SOP Reference	**SW-846 6010C, 7470A, 7471A/ CA-608, CA-611, CA-615**

QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Serial Dilution (SD) (does not apply to mercury)	One SD (5x) is performed for each batch of 20 samples of the same matrix.	If original sample result is at least 50x the instrument detection limit, the SD must agree within ± 10% of the original result.	Failure to meet the control limit shall be discussed in the case narrative, and elements will be flagged accordingly.	Analyst, Supervisor, Data Validator	Precision	Same as QC Acceptance Limits.
Post-Digestion Spike (does not apply to mercury)	For any element that fails in the MS where the native sample concentration was <4x the spike amount.	%R must be within 75-125% of the true value.	Discussed in the case narrative	Analyst, Laboratory Supervisor, Data Validator	Accuracy/ Bias	Same as QC Acceptance Limits.
Results between the MDL and LOQ	NA	Apply “J” qualifier to results detected between MDL and LOQ.	None.	Analyst, Supervisor, Data Validator	Precision	Same as QC Acceptance Limits.

Note that limits are updated periodically and may change from the issuance of the final SAP to the time data validation is performed. The limits used for validation will be the limits that are current at the time of analysis.

Matrix	**Soil, Groundwater, and Aqueous QC Samples**
Analytical Group	**TRPH**
Analytical Method/SOP Reference	**FL-PRO/ CA-333**

QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method Blank	One per preparatory batch of 20 or fewer samples.	The target analyte must be ≤ ½ LOQ.	Investigate source of contamination. Evaluate the samples and associated QC: i.e., if the blank results are >LOQ, then report sample results which are <LOQ or >10X the blank concentration. Otherwise, re-prepare a blank and samples >LOQ and <10X the blank.	Analyst, Supervisor, Data Validator	Bias/ Contamination	Same as QC Acceptance Limits.
Surrogate	Two per sample.	o-Terphenyl %R in waters must be between 82-142% in soils must be between 62-109. n-triacontane %Rs in soil and water must be between 70-130.	If surrogates %Rs are high and sample is <LOQ, then no corrective action is taken. If surrogates %Rs are low, then the affected samples are re-extracted and reanalyzed.	Analyst, Supervisor, Data Validator	Accuracy/ Bias	Same as QC Acceptance Limits.
LCS	One per preparatory batch of 20 or fewer samples.	%Rs must meet the limits provided in the Katahdin QC Limits table provided in Appendix D.	If an MS/MSD was performed and is acceptable, then narrate. If the LCS recovery is high, but the sample results are <LOQ, then narrate. Otherwise, re-extract blank and affected sample batch.	Analyst, Supervisor, Data Validator	Accuracy/ Bias	Same as QC Acceptance Limits.
MS/MSD	One per preparatory batch of 20 or fewer samples per matrix.	%Rs must meet the limits provided in the Katahdin QC Limits table provided in Appendix D. The RPD between MS and MSD should be ≤ 30%.	Evaluate the samples and associated QC and if the LCS results are acceptable, then narrate. If both the LCS and MS/MSD are unacceptable, then re-prepare the samples and QC.	Analyst, Supervisor, Data Validator	Accuracy/ Bias/ Precision	Same as QC Acceptance Limits.
Results between the MDL and LOQ	NA	Apply "J" qualifier to results detected between MDL and LOQ.	None.	Analyst, Supervisor, Data Validator	Precision	Same as QC Acceptance Limits.

Note that limits are updated periodically and may change from the issuance of the final SAP to the time data validation is performed. The limits used for validation will be the limits that are current at the time of analysis.

SAP Worksheet #29 – Project Documents and Records Table
(UFP-QAPP Manual Section 3.5.1)

Document	Where Maintained
Field Documents Field Logbook Field Sample Forms Chain of Custody Records Air Bills Sampling Instrument Calibration Logs Sampling Notes Photographs FTMR Forms This SAP HASP	Field documents will be maintained in the project file located in the Tetra Tech Jacksonville, Florida office.
Laboratory Documents Sample receipt, custody, and tracking record Equipment calibration logs Sample preparation logs Analysis Run logs Corrective Action forms Reported field sample results Reported results for standards, QC checks, and QC samples Extraction/clean-up records Raw data	Laboratory documents will be included in the hardcopy and portable documents format deliverables from the laboratory. Laboratory data deliverables will be maintained in the Tetra Tech Pittsburgh project file and in long-term data package storage at a third-party professional document storage firm. Electronic data results will be maintained in a database on a password protected Structured Query Language (SQL) server.
Assessment Findings Field Sampling Audit Checklist (if conducted) Analytical Audit Checklist (if conducted) Data Validation Memoranda (includes tabulated data summary forms)	All assessment documents will be maintained in the Tetra Tech Pittsburgh office.
Reports RI Report	All reports will be stored in hardcopy in the Tetra Tech Jacksonville project file and electronically in the server library.

SAP Worksheet #30 – Analytical Services Table
(UFP-QAPP Manual Section 3.5.2.3)

Matrix	Analytical Group	Sample Locations/ID Numbers	Analytical Method	Data Package Turnaround Time	Laboratory / Organization (name and address, contact person and telephone number)	Backup Laboratory/ Organization (name and address, contact person and telephone number)
Soil, Groundwater, and Aqueous QC Samples	VOCs	See Worksheet #18	SW-846 8260B	21 calendar days	Katahdin Analytical Services, Inc. 600 Technology Way Scarborough, Maine 04074 Ms. Kelly Perkins (207) 874-2400 Ext. 17 kperkins@katahdinlab.com	NA
	SVOCs (including low level PAHs by SIM)		SW-846 8270D/ 8270D SIM			
	PCBs		SW-846 8082A			
	Metals		SW-846 6010C, 7470A, and 7471A			
	TRPH		FL-PRO			

SAP Worksheet #31 – Planned Project Assessments Table
(UFP-QAPP Manual Section 4.1.1)

Assessment Type	**Frequency**	**Internal or External**	**Organization Performing Assessment**	**Person(s) Responsible for Performing Assessment** (title and organizational affiliation)	**Person(s) Responsible for Responding to Assessment Findings** (title and organizational affiliation)	**Person(s) Responsible for Identifying and Implementing Corrective Action** (title and organizational affiliation)	**Person(s) Responsible for Monitoring Effectiveness of Corrective Action** (title and organizational affiliation)
Laboratory System Audit[1]	Every two years	External	DoD ELAP Accrediting Body	DoD ELAP Accrediting Body Auditor	Laboratory QAM or Laboratory Manager, Katahdin	Laboratory QAM or Laboratory Manager, Katahdin	Laboratory QAM or Laboratory Manager, Katahdin

[1] Katahdin is DoD ELAP accredited and Florida NELAP accredited for all analytical groups and target analytes required for this project. The DoD ELAP and Florida NELAP accreditation documentation is included in Appendix D.

SAP Worksheet #32 – Assessment Findings and Corrective Action Responses Table
(UFP-QAPP Manual Section 4.1.2)

Assessment Type	**Nature of Deficiencies Documentation**	**Individual(s) Notified of Findings** (name, title, organization)	**Timeframe of Notification**	**Nature of Corrective Action Response Documentation**	**Individual(s) Receiving Corrective Action Response** (name, title, organization)	**Timeframe for Response**
Laboratory System Audit	Written audit report	Leslie Dimond, Laboratory QAM, Katahdin	Specified by DoD ELAP Accrediting Body	Letter	DoD ELAP Accrediting Body	Specified by DoD ELAP Accrediting Body

SAP Worksheet #33 – QA Management Reports Table
(UFP QAPP Manual Section 4.2)

Type of Report	Frequency (daily, weekly monthly, quarterly, annually, etc.)	Projected Delivery Date(s)	Person(s) Responsible for Report Preparation (title and organizational affiliation)	Report Recipient(s) (title and organizational affiliation)
Data Validation Report	Per SDG	Within 3 weeks of receipt of laboratory data package	DVM or designee, Tetra Tech	PM and project file, Tetra Tech
Major Analysis Problem Identification (Internal Tetra Tech Memorandum)	When persistent analysis problems are detected by Tetra Tech that may impact data usability	Immediately upon detection of problem (on the same day)	CLEAN QAM, Tetra Tech	PM, CLEAN QAM, Program Manager, and project file, Tetra Tech
Project Monthly Progress Report	Monthly for duration of project	Monthly	PM, Tetra Tech	Navy RPM, Navy; CLEAN QAM, Program Manager, and project file, Tetra Tech
Laboratory QA Report	When significant plan deviations result from unanticipated circumstances	Immediately upon detection of problem (on the same day)	Laboratory PM, Katahdin	PM and project file, Tetra Tech

SAP Worksheet #34 – Verification (Step I) Process Table
(UFP-QAPP Manual Section 5.2.1)

Verification Input	Description	Internal / External	Responsible for Verification (name, organization)
Chain-of-custody forms	The Tetra Tech FOL or designee will review and sign the chain-of-custody form to verify that all samples listed are included in the shipment to the laboratory and the sample information is accurate. The forms will be signed by the sampler and a copy will be retained for the project file, the Tetra Tech PM, and the Tetra Tech Data Validators. See Tetra Tech SOP SA-6.3.	Internal	Sampler and FOL, Tetra Tech
	The Laboratory Sample Custodian will review the sample shipment for completeness, integrity, and sign accepting the shipment. The Tetra Tech Data Validators will check that the chain-of-custody form was signed and dated by the Tetra Tech FOL or designee relinquishing the samples and also by the Laboratory Sample Custodian receiving the samples for analyses.	Internal/ External	1 - Laboratory Sample Custodian, Katahdin 2 - Data Validators, Tetra Tech
SAP Sample Tables/ Chain-of-Custody Forms	Verify that all proposed samples listed in the SAP tables have been collected.	Internal	FOL or designee, Tetra Tech
Sample Log Sheets	Verify that information recorded in the log sheets is accurate and complete.	Internal	FOL or designee, Tetra Tech
SAP/ Field Logs/ Analytical Data Packages	Ensure that all sampling SOPs were followed. Verify that deviations have been documented and measurement performance criteria (MPCs) have been achieved. Particular attention should be given to verify that samples were correctly identified, that sampling location coordinates are accurate, and that documentation establishes an unbroken trail of documented chain-of-custody from sample collection to report generation. Verify that the correct sampling and analytical methods/SOPs were applied. Verify that the sampling plan was implemented and carried out as written and that any deviations are documented.	Internal	PM or designee, Tetra Tech
SAP/ Laboratory SOPs/ Raw Data/ Applicable Control Limits Tables	Ensure that all laboratory SOPs were followed. Verify that the correct analytical methods/SOPs were applied. Establish that all method QC samples were analyzed and in control as listed in the analytical SOPs. If method QA is not in control, the Laboratory QAM will contact the Tetra Tech PM via telephone or e-mail for guidance prior to report preparation.	Internal	Laboratory QAM, Katahdin

Verification Input	Description	Internal / External	Responsible for Verification (name, organization)
SAP/ Chain-of-Custody Forms	Check that field QC samples listed in Worksheet #20 were collected as required.	Internal	FOL or designee, Tetra Tech
Analytical Data Packages	All analytical data packages will be verified internally for completeness by the laboratory performing the work. The Laboratory QAM will sign the case narrative for each data package.	Internal	Laboratory QAM, Katahdin

SAP Worksheet #35 – Validation (Steps IIa and IIb) Process Table
(UFP-QAPP Manual Section 5.2.2) (Figure 37, page 110 UFP-QAPP Manual) (Table 9 UFP-QAPP Manual)

Step IIa / IIb	Validation Input	Description	Responsible for Validation (name, organization)
IIa	SAP/ Sample Log Sheets	Verify that actual sample locations are correct and in accordance with the SAP proposed locations. Document in the final report any discrepancies that interfere with attainment of project objectives or that the project team believes could cause confusion for future data users.	PM, FOL, or designee, Tetra Tech
IIa	Chain-of-Custody Forms	Ensure that the custody and integrity of the samples was maintained from collection to analysis and the custody records are complete and any deviations are recorded. Review that the samples were shipped and stored at the required temperature and sample pH for chemically-preserved samples meet the requirements listed in Worksheet #19. Ensure that the analyses were performed within the holding times listed in Worksheet #19.	Project Chemist or Data Validators, Tetra Tech
IIa/IIb	SAP/ Laboratory Data Packages/ Electronic Data Deliverables (EDDs)	Ensure that the laboratory QC samples listed in Worksheet #28 were analyzed and that the MPCs listed in Worksheet #12 were met for all field samples and QC analyses. Check that specified field QC samples were collected and analyzed and that the analytical QC criteria set up for this project were met.	Project Chemist or Data Validators, Tetra Tech
		Check the field sampling precision by calculating the RPD for field duplicate samples. Check the laboratory precision by reviewing the RPD or percent difference values from laboratory duplicate analyses; MS/MSDs; and LCS/laboratory control sample duplicate (LCSD), if available. Ensure compliance with the methods and project MPCs accuracy goals listed in Worksheet #12.	
		Check that the laboratory recorded the temperature at sample receipt and the pH of the chemically preserved samples to ensure sample integrity from sample collection to analysis.	
		Review the chain-of-custody forms generated in the field to ensure that the required analytical samples have been collected, appropriate sample identifications have been used, and correct analytical methods have been applied. The Tetra Tech Data Validator will verify that elements of the data package required for validations are present, and if not, the laboratory will be contacted and the missing information will be requested. Validation will be performed as per Worksheet #36. Check that all data have been transferred correctly and completely to the final SQL database.	

Step IIa / IIb	Validation Input	Description	Responsible for Validation (name, organization)
IIb	SAP/ Laboratory Data Packages/ EDDs	Ensure that the project LOQs listed in Worksheet #15 were achieved.	Project Chemist or Data Validators, Tetra Tech
		Discuss the impact on reported MDLs due to matrix interferences or sample dilutions performed because of the high concentration of one or more other contaminants, on the other target compounds reported as non-detected. Document this usability issue and inform the Tetra Tech PM. Review and add PALs to the laboratory EDDs. Flag samples and notify the Tetra Tech PM of samples that exceed PSLs listed in Worksheet #15.	
		Ensure that all QC samples specified in the SAP were collected and analyzed and that the associated results were within prescribed SAP acceptance limits. Ensure that QC samples and standards prescribed in analytical SOPs were analyzed and within the prescribed control limits. If any significant QC deviations occur, the Laboratory QAM shall have contacted the Tetra Tech PM.	
		Summarize deviations from methods, procedures, or contracts in the Data Validation Report. Determine the impact of any deviation from sampling or analytical methods and SOPs requirements and matrix interferences effect on the analytical results. Qualify data results based on method or QC deviation and explain all the data qualifications. Print a copy of the project database qualified data depicting data qualifiers and data qualifiers codes that summarize the reason for data qualifications. Determine if the data met the MPCs and determine the impact of any deviations on the technical usability of the data.	

SAP Worksheet #36 – Analytical Data Validation (Steps IIa and IIb) Summary Table
(UFP-QAPP Manual Section 5.2.2.1) (Figure 37, page 110 UFP-QAPP Manual)

Step IIa / IIb	Matrix	Analytical Group	Validation Criteria	Data Validator (title and organizational affiliation)
IIa and IIb	Soil, Groundwater, and Aqueous QC Samples	VOCs, SVOCs (Including Low Level SVOCs and PAHs by SIM), PCBs, and TRPH by FL-PRO	Limited* data validation will be performed using criteria for SW-846 Methods 8260B, 8270D, 8270D SIM, 8082A, and FL-PRO listed in Worksheets #12, #15, #24, and #28 and the current DoD Quality Systems Manual (US DoD, 2009). If not included in the aforementioned, then the logic outlined in USEPA Contract Laboratory Program National Functional Guidelines for Organic Data Review EPA-540/R-99-008, October 1999 will be used to apply qualifiers to data.	Data Validation Specialist, Tetra Tech
IIa and IIb	Soil, Groundwater, and Aqueous QC Samples	Metals (Including Mercury)	Limited* data validation will be performed Using criteria for SW-846 Methods 6010C, 7470A, and 7471A listed in Worksheets #12, #15, #24, and #28 and the current DoD QSM. If not included in the aforementioned, then the logic outlined in USEPA Contract Laboratory Program National Functional Guidelines for Inorganic Data Review EPA 540-R-04-004, October 2004 will be used to apply qualifiers to data.	Data Validation Specialist, Tetra Tech

* Limited data validation. Limits the data review to specific review parameters (Data Completeness/Data Verification, Holding times, Calibrations, Blank Contamination, and Detection Limits) to determine gross deficiencies only. The limited data validation is best expressed as a review to preclude the possibility of false negatives and to eliminate false positives. Raw data are not evaluated and sample result verification is not conducted. A formal data validation report is prepared.

SAP Worksheet #37 – Usability Assessment
(UFP-QAPP Manual Section 5.2.3)

Data Usability Assessment

The usability of the data directly affects whether project objectives can be achieved. At a minimum the following characteristics will be evaluated. The results of these evaluations will be included in the project report. The characteristics will be evaluated for multiple concentration levels if the evaluator determines that this is necessary. To the extent required by the type of data being reviewed, the assessors will consult with other technically competent individuals to render sound technical assessments of these DQI characteristics:

Completeness

For each matrix that was scheduled to be sampled, the Tetra Tech FOL acting on behalf of the Partnering Team will prepare a table listing planned samples/analyses to collected samples/analyses. If deviations from the scheduled sample collection or analyses are identified the Tetra Tech PM and Project Risk Assessor will determine whether the deviations compromise the ability to meet project objectives. If they do, the Tetra Tech PM will consult with the Navy RPM and other Partnering Team members, as necessary (determined by the Navy RPM), to develop appropriate corrective actions.

Precision

The Tetra Tech Project Chemist acting on behalf of the Partnering Team will determine whether precision goals for field duplicates and laboratory duplicates were met. This will be accomplished by comparing duplicate results to precision goals identified in Worksheet #28. This will also include a comparison of field and laboratory precision with the expectation that laboratory duplicate results will be no less precise than field duplicate results. If the goals are not met, or data have been flagged as estimated (J qualifier), limitations on the use of the data will be described in the project report.

Accuracy

The Tetra Tech Project Chemist acting on behalf of the Partnering Team will determine whether the accuracy/bias goals were met for project data. This will be accomplished by comparing percent recoveries of LCS, LCSD, MS, MSD, and surrogate compounds to accuracy goals identified in Worksheet #28. This assessment will include an evaluation of field and laboratory contamination; instrument calibration variability; and analyte recoveries for surrogates, MS, and

Data Usability Assessment

LCSs. If the goals are not met, limitations on the use of the data will be described in the project report. Bias of the qualified results and a description of the impact of identified non-compliances on a specific data package or on the overall project data will be described in the project report.

Representativeness

A Tetra Tech Project Scientist identified by the Tetra Tech PM and acting on behalf of the Partnering Team will determine whether the data are adequately representative of intended populations, both spatially and temporally. This will be accomplished by verifying that samples were collected and processed for analysis in accordance with the SAP, by reviewing spatial and temporal data variations, and by comparing these characteristics to expectations. The usability report will describe the representativeness of the data for each matrix and analytical fraction. This will not require quantitative comparisons unless professional judgment of the project scientist indicates that a quantitative analysis is required.

Comparability

The Tetra Tech Project Chemist acting on behalf of the Partnering Team will determine whether the data generated under this project are sufficiently comparable to historical site data generated by different methods and for samples collected using different procedures and under different site conditions. This will be accomplished by comparing overall precision and bias among data sets for each matrix and analytical fraction. This will not require quantitative comparisons unless professional judgment of the Tetra Tech Project Chemist indicates that such quantitative analysis is required.

Sensitivity

The Tetra Tech Project Chemist acting on behalf of the Partnering Team will determine whether project sensitivity goals listed in Worksheet #15 are achieved. The overall sensitivity and quantitation limits from multiple data sets for each matrix and analysis will be compared. If sensitivity goals are not achieved, the limitations on the data will be described. The Tetra Tech Project Chemist will enlist the help of the Tetra Tech Risk Assessor to evaluate deviations from planned sensitivity goals.

Data Usability Assessment

Project Assumptions

The Tetra Tech PM and designated team members will evaluate whether project assumptions are valid. This will typically be a qualitative evaluation but may be supported by quantitative evaluations. The type of evaluation depends on the assumption being tested. Key assumptions relate to: direction of groundwater flow, stratigraphy (i.e, does the stratigraphy associated with PSC 45 consist of silty to clayey sands interbedded with layers of clay and sandy clay), data distributions (e.g., normal versus log-normal) and estimates of data variability.

Describe the evaluative procedures used to assess overall measurement error associated with the project:

After completion of the data validation, the data and data quality will be reviewed to determine whether sufficient data of acceptable quality are available for decision making. Statistical evaluations will include simple summary statistics for target analytes, such as maximum concentration, minimum concentration, average concentration, number of samples exhibiting non-detected results, number of samples exhibiting positive results, and the proportion of samples with detected and non-detected results. The Partnering Team members identified by the Tetra Tech PM will assess whether the data collectively support the attainment of project objectives. They will consider whether any missing or rejected data have compromised the ability to make decisions or to make the decisions with the desired level of confidence. The data will be evaluated to determine whether missing or rejected data can be compensated by other data. Although rejected data will generally not be used, there may be reason to use them in a weight of evidence argument, especially when they supplement data that have not been rejected. If rejected data are used, their use will be supported by technically defensible rationales.

The 95% UCL on the mean will be developed in accordance with the most current version of ProUCL (http://www.epa.gov/esd/tsc/software.htm). Duplicate results (original and duplicate) will not be averaged for the purpose of representing the range of concentrations. However, the average of the original and duplicate samples will be used to represent the concentration at a particular sampled location.

Data Usability Assessment

Identify the personnel responsible for performing the usability assessment:

The Tetra Tech PM, Project Chemist, FOL, and Project Scientist will be responsible for conducting the listed data usability assessments. The data usability assessment will be reviewed with the Navy RPM, the USEPA RPM, and the FDEP RPM. If deficiencies affecting the attainment of project objectives are identified, the review will take place either in a face to face meeting or a teleconference depending on the extent of identified deficiencies. If no significant deficiencies are identified, the data usability assessment will simply be documented in the project report and reviewed during the normal document review cycle.

Describe the documentation that will be generated during usability assessment and how usability assessment results will be presented so that they identify trends, relationships (correlations), and anomalies:

The data will be presented in tabular format, including data qualifications such as estimation (J, UJ) or rejection (R). Written documentation will support the non-compliance estimated or rejected data results. The project report will identify and describe the data usability limitations and suggest re-sampling or other corrective actions, if necessary.

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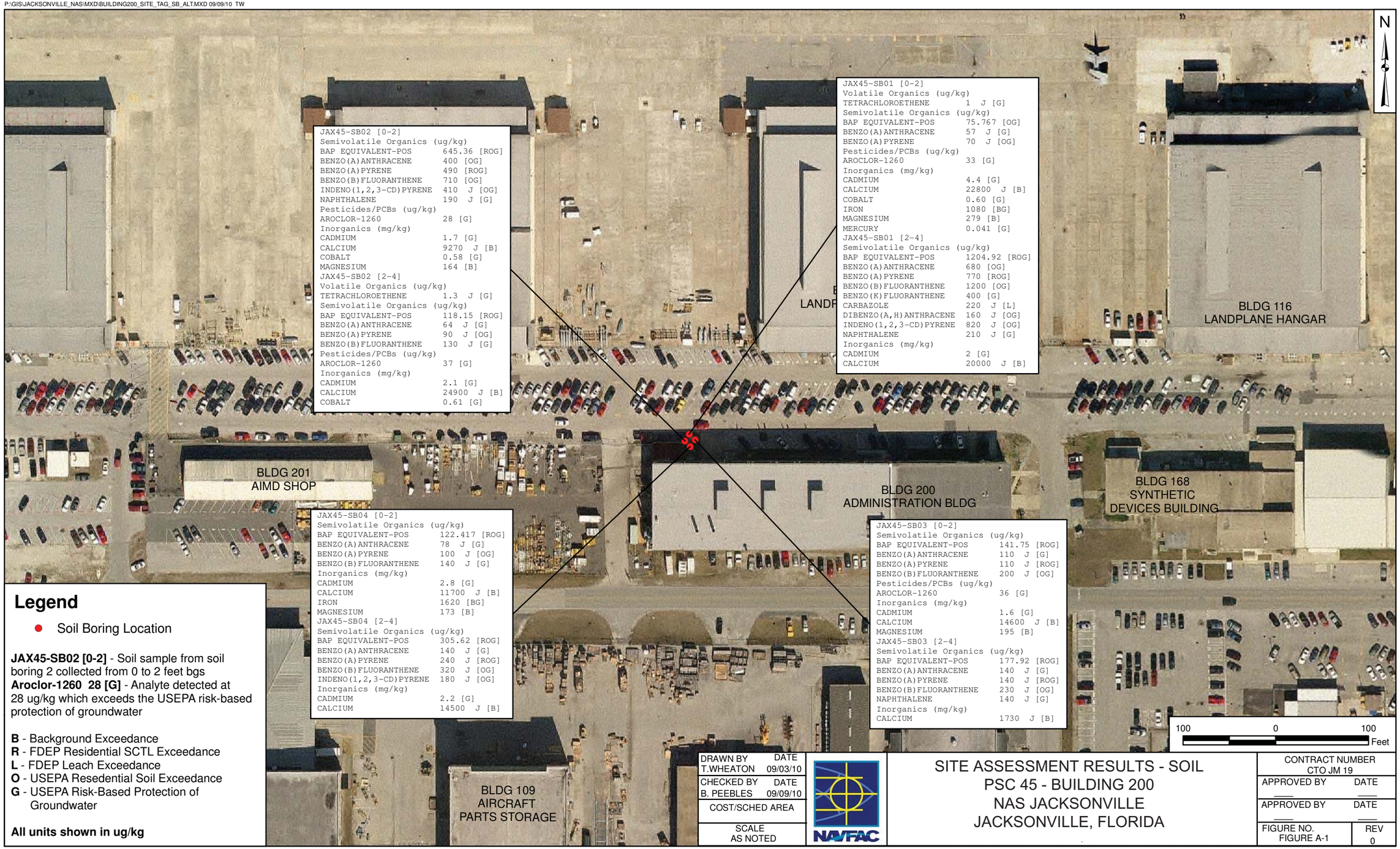
APPENDIX A

PSC 45 SITE ASSESSMENT GROUNDWATER AND SOIL RESULTS

SAMPLE DATE: AUGUST 2009

P:\GIS\JACKSONVILLE_NAS\MXD\BUILDING200_SITE_TAG_SB_ALT.MXD 09/09/10 TW



Legend

- Soil Boring Location

JAX45-SB02 [0-2] - Soil sample from soil boring 2 collected from 0 to 2 feet bgs
Aroclor-1260 28 [G] - Analyte detected at 28 ug/kg which exceeds the USEPA risk-based protection of groundwater

B - Background Exceedance
R - FDEP Residential SCTL Exceedance
L - FDEP Leach Exceedance
O - USEPA Resedential Soil Exceedance
G - USEPA Risk-Based Protection of Groundwater

All units shown in ug/kg

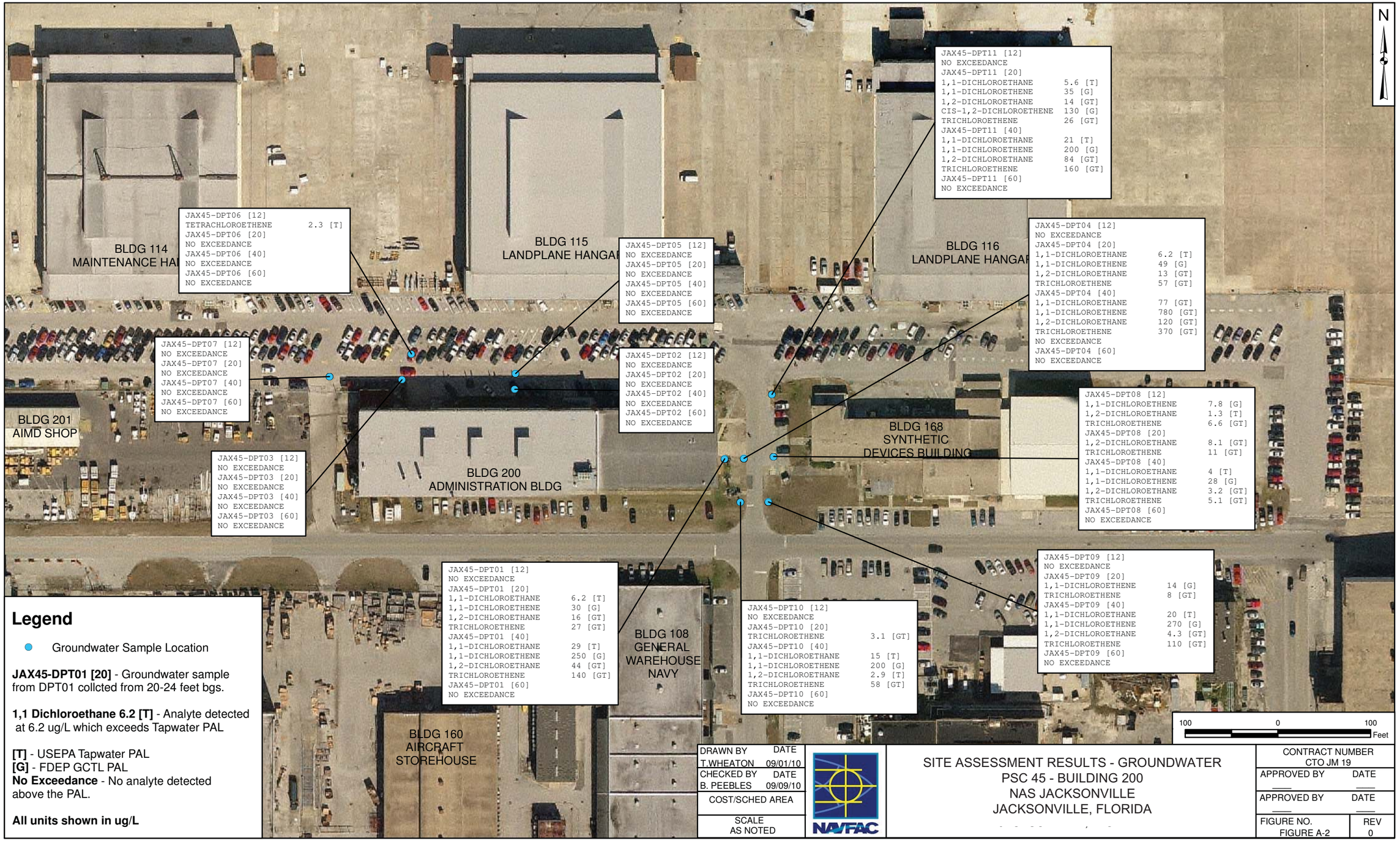
DRAWN BY T.WHEATON	DATE 09/03/10
CHECKED BY B. PEEBLES	DATE 09/09/10
COST/SCHED AREA	
SCALE AS NOTED	

NAVFAC

SITE ASSESSMENT RESULTS - SOIL
PSC 45 - BUILDING 200
NAS JACKSONVILLE
JACKSONVILLE, FLORIDA

CONTRACT NUMBER CTO JM 19	
APPROVED BY ___	DATE ___
APPROVED BY ___	DATE ___
FIGURE NO. FIGURE A-1	REV 0

P:\GIS\JACKSONVILLE_NAS\MXD\BUILDING200_GW_EXCEEDANCES_ALT.MXD 09/09/10 TW



Legend

- Groundwater Sample Location

JAX45-DPT01 [20] - Groundwater sample from DPT01 collcted from 20-24 feet bgs.

1,1 Dichloroethane 6.2 [T] - Analyte detected at 6.2 ug/L which exceeds Tapwater PAL

[T] - USEPA Tapwater PAL
[G] - FDEP GCTL PAL
No Exceedance - No analyte detected above the PAL.

All units shown in ug/L

DRAWN BY	DATE
T.WHEATON	09/01/10
CHECKED BY	DATE
B. PEEBLES	09/09/10
COST/SCHED AREA	
SCALE	
AS NOTED	

NAVFAC

SITE ASSESSMENT RESULTS - GROUNDWATER
PSC 45 - BUILDING 200
NAS JACKSONVILLE
JACKSONVILLE, FLORIDA

CONTRACT NUMBER	CTO JM 19
APPROVED BY	DATE
___	___
APPROVED BY	DATE
___	___
FIGURE NO.	REV
FIGURE A-2	0

APPENDIX B

NAS JACKSONVILLE BACKGROUND VALUES
PSLs FOR METALS

Table 4-1
Identification of Background Concentration - Surface Soil

Remedial Investigation and Feasibility Study, Operable Unit 1
Naval Air Station Jacksonville
Jacksonville, Florida

Parameter	Frequency of Detection[1]	Range of Detected Concentrations	Mean of Detected Concentrations[2]	Background Screening Concentration[3]
Volatiles (µg/kg)				
Acetone	6/10	3 - 11	6.7	NA
Semivolatiles (µg/kg)				
Anthracene	1/10	37 - 37	37	NA
Benzo(a)Anthracene	2/10	27 - 250	139	NA
Benzo(a)Pyrene	3/10	29 - 150	71	NA
Benzo(b)Fluoranthene	3/10	26 - 330	140	NA
Benzo(g,h,i)Perylene	3/10	43 - 80	57.3	NA
Benzo(k)Fluoranthene	3/10	20 - 100	49.3	NA
Carbazole	1/10	46 - 46	46	NA
Chrysene	3/10	24 - 350	138	NA
Di-n-Butylphthalate	1/10	355 - 355	355	NA
Dibenz(a,h)Anthracene	2/10	18 - 31	24.5	NA
Fluoranthene	3/10	20 - 390	147	NA
Indeno(1,2,3-cd)Pyrene	3/10	41 - 88	57	NA
Phenanthrene	1/10	36 - 36	36	NA
Phenol	1/10	19 - 19	19	NA
Pyrene	3/10	28 - 430	163	NA
bis(2-Ethylhexyl)Phthalate	1/10	18 - 18	18	NA
Pesticides and PCBs (µg/kg)				
4,4-DDD	1/10	2.7 - 2.7	2.7	NA
4,4-DDE	4/10	1.8 - 12	4.6	NA
4,4-DDT	3/10	2.2 - 18	8.2	NA
Aroclor-1260	1/10	26 - 26	26	NA
Dieldrin	2/10	0.43 - 97	48.7	NA
alpha-Chlordane	1/10	0.25 - 0.25	0.25	NA
gamma-Chlordane	2/10	0.37 - 0.81	0.59	NA
Dioxins (µg/kg)				
1,2,3,4,6,7,8-HpCDD	1/3	0.0614 - 0.0614	0.0614	NA
OCDD	2/3	0.211 - 0.517	0.364	NA
Inorganics (mg/kg)				
Aluminum	10/10	31.8 - 1,710	670	1,340
Arsenic	7/10	0.29 - 0.6	0.4	0.8
Barium	10/10	1.1 - 12.7	5.6	11.2
Calcium	10/10	48.2 - 6,200	1,180	2,360
Chromium	7/10	1.5 - 4.6	3.3	6.6
Copper	3/10	1.7 - 5.2	2.9	5.8

See notes at end of table.

Table 4-1 (Continued)
Identification of Background Concentration - Surface Soil

Remedial Investigation and Feasibility Study, Operable Unit 1
Naval Air Station Jacksonville
Jacksonville, Florida

Parameter	Frequency of Detection[1]	Range of Detected Concentrations	Mean of Detected Concentrations[2]	Background Screening Concentration[3]
Inorganics (mg/kg)				
Cyanide	3/7	0.18 - 0.22	0.2	0.4
Iron	10/10	124 - 928	426	852
Lead	10/10	1.2 - 26.6	7.2	14.4
Magnesium	9/10	15.9 - 154	49.9	99.8
Manganese	10/10	1.4 - 37.4	9	18
Nickel	5/10	2.8 - 14.7	5.5	11
Sodium	8/10	103 - 221	144	288
Thallium	1/10	0.21 - 0.21	0.21	0.42
Vanadium	8/9	0.58 - 4.6	1.9	3.8
Zinc	8/10	3.8 - 16.1	7.6	15.2
Radioisotopes (pCi/g)[4]				
Actinium-228	10/10	0.652 - 1.46	1	2
Bismuth-210	4/10	1.4 - 2.58	1.9	3.8
Bismuth-214	10/10	0.437 - 1.02	0.71	1.42
Cesium-137	8/10	0.0301 - 0.527	0.14	0.28
Lead-212	10/10	0.325 - 0.923	0.63	1.26
Lead-214	10/10	0.412 - 0.991	0.67	1.34
Potassium-40	10/10	0.8 - 4.335	2.6	5.2
Radium-223	4/10	0.709 - 1.56	1.1	2.2
Radium-224	7/10	0.82 - 2.14	1.5	3.0
Radium-228	10/10	0.652 - 1.46	1	2
Thallium-208	10/10	0.175 - 0.529	0.33	0.66
Thorium-231	5/10	0.069 - 0.145	0.12	0.24
Thorium-232	9/10	0.724 - 1.46	1.1	2.2
Thorium-234	2/10	0.595 - 4.24	2.4	4.8
Uranium-234	2/10	2.23 - 2.38	2.3	4.6
Uranium-238	2/10	2.23 - 2.38	2.3	4.6

[1] Frequency of detection is the number of samples in which the analyte was detected divided by the total number of samples analyzed. The samples are identified in Section 4.2.1.7 and Appendix P-4.
[2] The average of detected concentrations is the arithmetic mean of all samples in which the analyte was detected. It does not include those samples in which the analyte was not detected.
[3] Two times the mean for inorganic analytes and radionuclides. Values of organic compounds detected in background surface soil samples are considered on a case-by-case basis in the evaluation of "site" samples.
[4] The surface soil background screening concentration for radium-226, calculated from lead-214 concentrations, has been determined at 1.3 pCi/g.

Notes: µg/kg = micrograms per kilogram.
NA = not applicable.
PCBs = polychlorinated biphenyls.
DDD = dichlorodiphenyldichloroethane.
DDE = dichlorodiphenyldichloroethene.
DDT = dichlorodiphenyltrichloroethane.
HpCDD = heptachlorodibenzo-p-dioxin.
OCDD = octachlorodibenzodioxin.
mg/kg = milligrams per kilogram.
pCi/g = picocuries per gram.

Table 4-2
Identification of Background Concentration - Subsurface Soil

Remedial Investigation and Feasibility Study, Operable Unit 1
Naval Air Station Jacksonville
Jacksonville, Florida

Parameter	Frequency of Detection[1]	Range of Detected Concentrations	Mean of Detected Concentrations[2]	Background Screening Concentration[3]
Volatiles (µg/kg)				
Acetone	4/11	4 - 11	7.25	NA
Semivolatiles (µg/kg)				
Benzo(a)Pyrene	1/11	21 - 21	21	NA
Benzo(b)Fluoranthene	1/11	34 - 34	34	NA
Benzo(g,h,i)Perylene	1/11	29 - 29	29	NA
bis(2-Ethylhexyl)Phthalate	6/11	54 - 170	90.33	NA
Di-n-Butylphthalate	1/11	460 - 460	460	NA
Indeno(1,2,3-cd)Pyrene	1/11	23 - 23	23	NA
Phenol	4/11	20 - 24	21.5	NA
Pesticides and PCBs (µg/kg)				
4,4-DDE	1/11	0.29 - 0.29	0.29	NA
4,4-DDT	1/11	1.7 - 1.7	1.7	NA
alpha-Chlordane	2/11	0.37 - 0.45	0.41	NA
gamma-Chlordane	2/10	0.41 - 0.55	0.48	NA
Methoxychlor	1/11	1.2 - 1.2	1.2	NA
Inorganics (mg/kg)				
Aluminum	10/10	373 - 7,620	3,411.6	6,823.2
Arsenic	6/10	0.41 - 2.0	0.74	1.48
Barium	10/10	2.0 - 20.9	10.4	20.8
Beryllium	2/10	0.24 - 0.25	0.245	0.49
Calcium	10/10	44.8 - 1,200	334.15	668.3
Chromium	9/10	2.9 - 12.3	7.056	14.1
Iron	10/10	105 - 15600	2909.1	5818.2
Lead	10/10	1.5 - 5.6	3.23	6.46
Magnesium	8/10	104 - 700	250.125	500.25
Manganese	10/10	1.5 - 7.2	3.45	6.90
Potassium	3/10	187 - 252	225.33	450.67
Sodium	9/10	117 - 342	171.556	343.10
Zinc	9/10	4.1 - 12.8	7.244	14.49
Radionuclides (pCi/g)[4]				
Actinium-228	8/10	0.466 - 1.95	1.201	2.40
Bismuth-210	3/10	0.444 - 2.03	1.241	2.48
Bismuth-214	9/10	0.642 - 1.58	0.936	1.87
Lead-212	10/10	0.237 - 1.17	0.671	1.34
Lead-214	10/10	0.212 - 1.34	0.744	1.49

See notes at end of table.

Table 4-2 (Continued)
Identification of Background Concentration - Subsurface Soil

Remedial Investigation and Feasibility Study, Operable Unit 1
Naval Air Station Jacksonville
Jacksonville, Florida

Parameter	Frequency of Detection[1]	Range of Detected Concentrations	Mean of Detected Concentrations[2]	Background Screening Concentration[3]
Radionuclides (pCi/g)				
Potassium-40	8/10	2.12 - 8.76	5.57	11.14
Radium-223	3/10	0.286 - 1.56	0.9	1.80
Radium-224	6/10	1.13 - 2.31	1.585	3.17
Radium-228	8/10	0.429 - 1.95	1.216	2.43
Thallium-208	10/10	0.113 - 0.511	0.328	0.66
Thorium-231	6/10	0.044 - 0.240	0.145	0.29
Thorium-232	7/10	0.429 - 1.95	1.171	2.34
Thorium-234	5/10	2.96 - 4.53	3.484	6.98
Uranium-234	5/10	1.6 - 2.52	1.962	3.92
Uranium-238	5/10	1.6 - 2.52	1.962	3.92
Vanadium	9/10	0.99 - 16.7	7.288	14.58

[1] Frequency of detection is the number of samples in which the analyte was detected divided by the total number of samples analyzed. The samples are identified in Section 4.2.2.6 and Appendix P-4.
[2] The average of detected concentrations is the arithmetic mean of all samples in which the analyte was detected. It does not include those samples in which the analyte was not detected.
[3] Two times the mean for inorganic analytes and radionuclides. Values of organic compounds detected in background surface soil samples are considered on a case-by-case basis in the evaluation of "site" samples.
[4] The subsurface soil background screening concentration for radium-226, calculated from lead-214 concentrations, has been determined at 1.5 pCi/g.

Notes: µg/kg = micrograms per kilogram.
NA = not applicable.
PCBs = polychlorinated biphenyls.
DDE = dichlorodiphenyldichloroethene.
DDT = dichlorodiphenyltrichloroethane.
mg/kg = milligrams per kilogram.
pCi/g = picocuries per gram.

Table 4-3
List of Soil Sampling Locations

Remedial Investigation and Feasibility Study, Operable Unit 1
Naval Air Station Jacksonville
Jacksonville, Florida

Soil Locations Inside Boundaries of Presumptive Remedy

SL001	SL027	SL045	SL099
SL002	SL028	SL046	SL100
SL003	SL029	SL047	SL101
SL009	SL030	SL048	SL102
SL010	SL032	SL049	SL103
SL011	SL033	SL050	SL27001
SL012	SL034	SL051	SL27002
SL013	SL035	SL052	SL27003
SL014	SL036	SL064	SL27004
SL016	SL037	SL072	SL27005
SL017	SL038	SL083	SL27006
SL018	SL039	SL088	SL27007
SL019	SL040	SL091	SL27008
SL022	SL041	SL093	SL27009
SL023	SL042	SL094	SL27010
SL024	SL043	SL097	SL27011
SL025	SL044	SL098	

Soil Locations Outside Boundaries of Presumptive Remedy

SL004	SL063	SL087	SL119
SL005	SL065	SL089	SL120
SL006	SL066	SL090	SL121
SL007	SL067	SL092	SL122
SL008	SL068	SL095	SL123
SL015	SL069	SL096	SL124
SL020	SL070	SL104	SL125
SL021	SL071	SL105	SL126
SL026	SL073	SL106	SL127
SL031	SL074	SL107	U1DSMW100
SL053	SL075	SL108	U1DSMW102
SL054	SL076	SL109	U1DSMW104
SL055	SL077	SL110	U1DSMW106
SL056	SL078	SL111	U1DSMW108
SL057	SL079	SL112	U1DSMW88
SL058	SL080	SL113	U1DSMW90
SL059	SL081	SL114	U1DSMW94
SL060	SL082	SL115	U1DSMW96
SL061	SL084	SL116	U1DSMW98
SL062	SL085	SL117	U1SSMW93
	SL086	SL118	U1SSMW97
			U1SSMW99

Table 4-4
Identification of Background Concentrations - Surface Water

Remedial Investigation and Feasibility Study, Operable Unit 1
Naval Air Station Jacksonville
Jacksonville, Florida

Parameter	Frequency of Detection[1]	Range of Detected Concentrations	Mean of Detected Concentrations[2]	Background Screening Concentration[3]
Inorganics (µg/ℓ)				
Arsenic	3/4	0.7 - 2.9	1.6	3.2
Barium	4/4	29.2 - 70.2	41.5	83
Calcium	4/4	7,320 - 34,200	19,555	39,110
Copper	4/4	2.1 - 7.1	3.8	7.6
Cyanide	3/4	0.6 - 3.1	1.5	3
Iron	4/4	362 - 1,920	1,218	2,436
Lead	4/4	0.8 - 8.7	3.3	6.6
Magnesium	4/4	1,800 - 5,090	3,063	6,126
Manganese	4/4	6.1 - 28.9	19.8	39.6
Potassium	4/4	453 - 1,530	896	1,792
Sodium	4/4	7,770 - 14,400	10,435	20,870
Vanadium	4/4	2 - 3.4	2.8	5.6
Zinc	4/4	14.8 - 38.7	23.2	46.4
Radionuclides (pCi/ℓ)				
Bismuth-214	1/4	11.2 - 11.2	11.2	(*)
Thorium-234	1/4	158 - 158	158	(*)
Dissolved Inorganics (µg/ℓ)				
Diss. Aluminum	4/4	32.5 - 301	211	422
Diss. Arsenic	4/4	0.9 - 2.7	1.5	3.0
Diss. Barium	4/4	28.8 - 48.1	35.3	70.6
Diss. Cadmium	1/4	0.73 - 0.73	0.73	1.46
Diss. Calcium	4/4	7,050 - 32,500	19,013	38,026
Diss. Copper	3/4	1.6 - 6.2	3.1	6.2
Diss. Iron	4/4	232 - 1,090	601	1,202
Diss. Lead	2/4	0.9 - 1.5	1.2	2.4
Diss. Magnesium	4/4	1,780 - 4,930	3,013	6,026
Diss. Manganese	4/4	6.8 - 29.2	17.8	35.6
Diss. Potassium	4/4	615 - 1,430	940	1,880
Diss. Sodium	4/4	7,760 - 14,300	10,410	20,820
Diss. Vanadium	1/4	4.3 - 4.3	4.3	8.6
Diss. Zinc	4/4	14.5 - 21.1	17.8	35.6

[1] Frequency of detection is the number of samples in which the analyte was detected divided by the total number of samples analyzed. The four samples used are taken from SW/SD58, SW/SD59, SW/SD60 and SW/SD62.
[2] The average of detected concentrations is the mean of all samples in which the analyte was detected. It does not include those samples in which the analyte was not detected.
[3] Two times the mean for inorganic analytes and radiological parameters.
(*) Background screening concentrations for radionuclides were not developed for this set.

Notes: µg/ℓ = micrograms per liter.
pCi/ℓ = picocuries per liter.

Table 4-5
Identification of Background Concentrations - Sediment

Remedial Investigation and Feasibility Study, Operable Unit 1
Naval Air Station Jacksonville
Jacksonville, Florida

Chemical	Frequency of Detection[1]	Range of Detected Concentrations	Mean of Detected Concentrations[2]	Background Screening Concentration[3]
Volatiles (µg/kg)				
2-Butanone	1/4	8 - 8	8	NA
Acetone	3/4	24 - 35	28.7	NA
Semivolatiles (µg/kg)				
Anthracene	1/4	84 - 84	84	NA
Benzo(a)Anthracene	1/4	470 - 470	470	NA
Benzo(a)Pyrene	1/4	480 - 480	480	NA
Benzo(b)Fluoranthene	1/4	540 - 540	540	NA
Benzo(g,h,i)Perylene	1/4	90 - 90	90	NA
Benzo(k)Fluoranthene	1/4	370 - 370	370	NA
Chrysene	1/4	540 - 540	540	NA
Dibenz(a,h)Anthracene	1/4	80 - 80	80	NA
Fluoranthene	1/4	1,300 - 1,300	1,300	NA
Indeno(1,2,3-cd)Pyrene	1/4	180 - 180	180	NA
Phenanthrene	1/4	590 - 590	590	NA
Pyrene	1/4	1,100 - 1,100	1,100	NA
Pesticides and PCBs (µg/kg)				
4,4-DDD	1/4	51 - 51	51	NA
4,4-DDE	2/4	3.1 - 170	86.6	NA
Inorganics (mg/kg)				
Aluminum	4/4	239 - 1,220	595	1,190
Antimony	1/4	4.6 - 4.6	4.6	9.2
Arsenic	3/4	0.2 - 0.97	0.63	1.26
Barium	4/4	2.2 - 9.6	4.9	9.8
Beryllium	1/4	0.24 - 0.24	0.24	0.48
Cadmium	1/4	0.3 - 0.3	0.3	0.6
Calcium	4/4	124 - 8,660	3,234	6,468
Chromium	3/4	0.73 - 2.9	1.9	3.8
Cobalt	1/4	1.9 - 1.9	1.9	3.8
Copper	3/4	2.6 - 4.2	3.5	7
Cyanide	3/4	0.06 - 0.11	0.08	0.16
Iron	4/4	560 - 2,290	1,150	2,300
Lead	4/4	2 - 12.3	7.2	14.4
Magnesium	4/4	25.2 - 110	65.5	131
Manganese	4/4	1.5 - 4.9	3.4	6.8
Mercury	1/4	0.05 - 0.05	0.05	0.1

See notes at end of table.

Table 4-5 (Continued)
Identification of Background Concentrations - Sediment

Remedial Investigation and Feasibility Study, Operable Unit 1
Naval Air Station Jacksonville
Jacksonville, Florida

Chemical	Frequency of Detection[1]	Range of Detected Concentrations	Mean of Detected Concentrations[2]	Background Screening Concentration[3]
Inorganics (mg/kg)				
Nickel	3/4	2.9 - 3.4	3.1	6.2
Potassium	1/4	109 - 109	109	218
Selenium	1/4	0.21 - 0.21	0.21	0.42
Sodium	3/4	239 - 260	249	498
Thallium	1/4	0.19 - 0.19	0.19	0.38
Vanadium	3/4	0.77 - 4.6	2.6	5.2
Zinc	4/4	2.6 - 18.8	9.2	18.4
Radionuclides (pCi/g)				
Actinium-228	4/4	0.64 - 0.887	0.8	1.6
Bismuth-212	2/4	1.06 - 1.34	1.2	2.4
Bismuth-214	4/4	0.418 - 0.668	0.54	1.08
Cesium-137	2/4	0.0953 - 0.14	0.12	0.24
Lead-212	4/4	0.378 - 0.816	0.52	1.04
Lead-214	1/4	0.447 - 0.447	0.45	0.90
Potassium-40	1/4	3.66 - 3.66	3.7	7.4
Thallium-208	4/4	0.188 - 0.286	0.24	0.48
Uranium-235	1/4	0.125 - 0.125	0.13	0.26

[1] Frequency of detection is the number of samples in which the analyte was detected divided by the total number of samples analyzed. The samples used are taken from SW/SD58, SW/SD59, SW/SD60, and SW/SD62.
[2] The average of detected concentrations is the mean of all samples in which the analyte was detected. It does not include those samples in which the analyte was not detected.
[3] Two times the mean for inorganic analytes and radiological parameters.

Notes: µg/kg = micrograms per kilogram.
NA = not applicable.
PCBs = polychlorinated biphenyls.
DDD = dichlorodiphenyldichloroethane.
DDE = dichlorodiphenyldichloroethene.
mg/kg = milligrams per kilogram.
pCi/g = picocuries per gram.

Table 4-6
Identification of Background Screening Concentration - Groundwater

Remedial Investigation and Feasibility Study, Operable Unit 1
Naval Air Station Jacksonville
Jacksonville, Florida

Parameter	Frequency of Detection[1]	Range of Detected Concentrations	Mean of Detected Concentrations[2]	Background Screening Concentration[3]
Volatiles (µg/ℓ)				
Carbon Disulfide	5/42	1 - 7	3	NA
Chloroform	1/42	2 - 2	2	NA
Chloromethane	2/42	1 - 3	2	NA
Xylene (total)	1/42	1 - 1	1	NA
Semivolatiles (µg/ℓ)				
Diethylphthalate	1/42	3 - 3	3	NA
Phenol	1/42	1 - 1	1	NA
bis(2-Ethylhexyl)Phthalate	18/42	0.6 - 64	6.1	NA
Pesticides and PCBs (µg/ℓ)				
4,4'-DDE	1/42	0.006 - 0.006	0.01	NA
Dieldrin	1/42	*0.016 - *0.016	0.02	NA
Inorganics (µg/ℓ)				
Aluminum	42/42	146 - 451,000	73,659	147,318
Antimony	2/40	20.2 - *22.7	21.5	43
Arsenic	34/42	*1.05 - 14.2	6.6	13.2
Barium	42/42	19.3 - 3,160	308	616
Beryllium	30/42	*0.335 - 30	4.1	8.2
Cadmium	12/42	0.78 - 8.8	4.1	8.2
Calcium	42/42	2,300 - 163,000	29,533	59,066
Chromium	36/42	*2.35 - 542	104	208
Cobalt	26/42	3.5 - 57.8	11.3	22.6
Copper	30/42	3.2 - 78.5	20.2	40.4
Cyanide	4/42	1.8 - 2.5	2.2	4.4
Iron	42/42	255 - 187,000	34,146	68,292
Lead	36/42	0.5 - 136	22.9	45.8
Magnesium	42/42	3,340 - 36,700	9,658	19,316
Manganese	42/42	7.4 - 1,240	102	204
Mercury	18/42	0.14 - 2.1	0.49	0.98
Nickel	21/42	9.6 - 174	37.4	74.8
Potassium	41/42	902 - 17,700	4,519	9,038
Selenium	9/42	*0.56 - 47.9	6.9	13.8
Silver	2/42	4.2 - *5.1	4.7	9.4
Sodium	42/42	790 - 29,000	12,313	24,626
Vanadium	37/42	*2.625 - *728.5	147	294
Inorganics (µg/ℓ)				
Zinc	42/42	6.6 - 261	86.6	173.2

See notes at end of table.

Table 4-6 (Continued)
Identification of Background Screening Concentration - Groundwater

Remedial Investigation and Feasibility Study, Operable Unit 1
Naval Air Station Jacksonville
Jacksonville, Florida

Parameter	Frequency of Detection[1]	Range of Detected Concentrations	Mean of Detected Concentrations[2]	Background Screening Concentration[3]
Radioisotope ($\mu g/\ell$)				
Actinium-228	2/42	*11.7 - 21.8	16.8	33.6
Bismuth-214	3/42	*5.3 - 11.6	7.5	15
Lead-214	3/42	*6.7 - 18.1	13.3	26.6
Potassium-40	3/42	60.1 - 138	92.6	185.6
Radium-224	6/42	*54.5 - 105	88.4	176.8
Thallium-208	3/42	*3.7 - 7.3	5.7	11.4
Diss. Aluminum	34/42	8 - 74,200	8,905	NA
Diss. Antimony	9/42	12.8 - *30.05	19.6	NA
Diss. Arsenic	16/42	*0.6 - 6.6	3.2	NA
Diss. Barium	42/42	*9.9 - *250	64.1	NA
Diss. Beryllium	10/42	*0.43 - 3.2	1.3	NA
Diss. Cadmium	4/42	1 - 6.2	3.7	NA
Diss. Calcium	42/42	1,130 - *99,300	23,232	NA
Diss. Chromium	16/42	2.5 - 75.8	23.7	NA
Diss. Cobalt	8/42	*2.75 - *6.4	4.8	NA
Diss. Copper	22/42	1.1 - 12.7	5.3	NA
Diss. Iron	41/42	11.8 - *27,800	4,509	NA
Diss. Lead	21/42	0.6 - 18.9	4.1	NA
Diss. Magnesium	42/42	1,030 - *15,800	4,773	NA
Diss. Manganese	41/42	*2.65 - 134	35.4	NA
Diss. Mercury	1/42	0.1 - 0.1	0.1	NA
Diss. Nickel	4/42	*10.3 - 19.5	13.2	NA
Diss. Potassium	42/42	585 - *5,770	1,912	NA
Diss. Selenium	2/42	1.2 - 4.1	2.7	NA
Diss. Sodium	42/42	2,070 - 31,200	12,410	NA
Diss. Thallium	1/42	1 - 1	1	NA
Diss. Vanadium	24/42	*2.625 - *105.55	25.6	NA
Diss. Zinc	39/42	6.2 - 134	35.1	NA

[1] Frequency of detection is the number of samples in which the analyte was detected divided by the total number of samples analyzed. The samples analyzed are identified in Table R-4.7.
[2] The mean of detected concentrations is the mean of all samples in which the analyte was detected. It does not include those samples in which the analyte was not detected.
[3] Two times the mean for inorganic analytes.
[4] The groundwater background screening concentration for radium-226, calculated from lead-214 concentrations, has been determined at 26.6 pCi/ℓ.
* Value is the average of a sample and its duplicate.

Notes: $\mu g/\ell$ = micrograms per liter.
NA = not applicable.
PCBs = polychlorinated biphenyls.
DDE = dichlorodiphenyldichloroethene.

APPENDIX C

FIELD STANDARD OPERATING PROCEDURES AND FIELD DATA SHEETS

Tetra Tech NUS, Inc.

EQUIPMENT CALIBRATION LOG

PROJECT NAME : ______________________ INSTRUMENT NAME/MODEL: ______________________

SITE NAME: ______________________ MANUFACTURER: ______________________

PROJECT No.: ______________________ SERIAL NUMBER: ______________________

Date of Calibration	Instrument I.D. Number	Person Performing Calibration	Instrument Settings		Instrument Readings		Calibration Standard (Lot No.)	Remarks and Comments
			Pre-calibration	Post-calibration	Pre-calibration	Post-calibration		

Tetra Tech NUS, Inc.

DAILY ACTIVITIES RECORD

PROJECT NAME: _____ **PROJECT NUMBER:** _____

CLIENT: _____ **LOCATION:** _____

DATE: _____ **ARRIVAL TIME:** _____

Tt NUS PERSONNEL: _____ **DEPARTURE TIME:** _____

CONTRACTOR: _____ **DRILLER:** _____

ITEM	QUANTITY ESTIMATE	QUANTITY TODAY	PREVIOUS TOTAL QUANTITY	CUMULATIVE QUANTITY TO DATE

COMMENTS: _____

_____

_____

_____

APPROVED BY:

_____ _____

Tt NUS REPRESENTATIVE DRILLER

DATE: _____

Tetra Tech NUS, Inc.

GROUNDWATER SAMPLE LOG SHEET

Project Site Name: _____
Project No.: _____

[] Domestic Well Data
[] Monitoring Well Data
[] Other Well Type: _____
[] QA Sample Type: _____

Sample ID No.: _____
Sample Location: _____
Sampled By: _____
C.O.C. No.: _____
Type of Sample:
[] Low Concentration
[] High Concentration

SAMPLING DATA:

Date: / Time:	**Color** (Visual)	**pH** (S.U.)	**S.C.** (mS/cm)	**Temp.** (^{0}C)	**Turbidity** (NTU)	**DO** (mg/l)	**Salinity** (%)	Other
Method:								

PURGE DATA:

Date:	**Volume**	**pH**	**S.C.**	**Temp.**	**Turbidity**	**DO**	**Salinity**	Other
Method:								
Monitor Reading (ppm):								
Well Casing Diameter & Material Type:								
Total Well Depth (TD):								
Static Water Level (WL):								
One Casing Volume(gal/L):								
Start Purge (hrs):								
End Purge (hrs):								
Total Purge Time (min):								
Total Vol. Purged (gal/L):								

SAMPLE COLLECTION INFORMATION:

Analysis	**Preservative**	**Container Requirements**	**Collected**

OBSERVATIONS / NOTES:

Circle if Applicable:

MS/MSD | **Duplicate ID No.:**

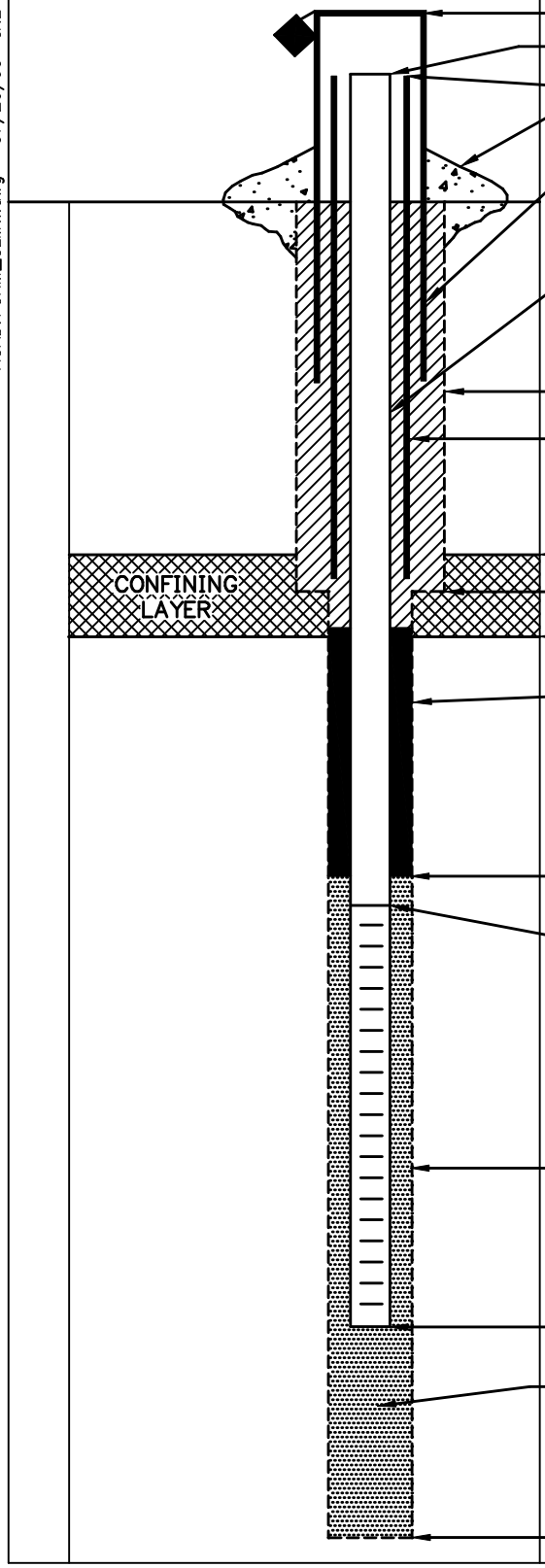
Signature(s):

WELL NO.: _____

Tetra Tech NUS, Inc.

CONFINING LAYER
MONITORING WELL SHEET

PROJECT _____	LOCATION _____	DRILLER _____
PROJECT NO. _____	BORING _____	DRILLING METHOD _____
DATE BEGUN _____	DATE COMPLETED _____	
FIELD GEOLOGIST _____		DEVELOPMENT METHOD _____
GROUND ELEVATION _____	DATUM _____	



ELEVATION/HEIGHT OF TOP OF SURFACE CASING: ____/____

ELEVATION/HEIGHT OF TOP OF RISER PIPE: ____/____

ELEVATION/HEIGHT OF TOP OF PERM. CASING: ____/____

TYPE OF SURFACE SEAL: _____

_____

I.D. OF SURFACE CASING: _____

TYPE OF SURFACE CASING: _____

_____

RISER PIPE I.D.: _____

TYPE OF RISER PIPE: _____

_____

BOREHOLE DIAMETER: _____

PERM. CASING I.D.: _____

TYPE OF CASING AND BACKFILL: _____

_____

ELEVATION/DEPTH TOP CONFINING LAYER: ____/____

ELEVATION/DEPTH BOTTOM OF CASING: ____/____

ELEVATION/DEPTH BOTTOM CONFINING LAYER: ____/____

ELEVATION/DEPTH TOP OF SEAL: ____/____

TYPE OF SEAL: _____

_____

DEPTH TOP OF SAND PACK: _____

ELEVATION/DEPTH TOP OF SCREEN: ____/____

TYPE OF SCREEN: _____

_____

TYPE OF SAND PACK: _____

_____

BOREHOLE DIA. BELOW CASING: _____

ELEVATION/DEPTH BOTTOM OF SCREEN: ____/____

ELEVATION/DEPTH BOTTOM OF SAND PACK: ____/____

BACKFILL MATERIAL BELOW SAND: _____

_____

ELEVATION/DEPTH OF HOLE: ____/____

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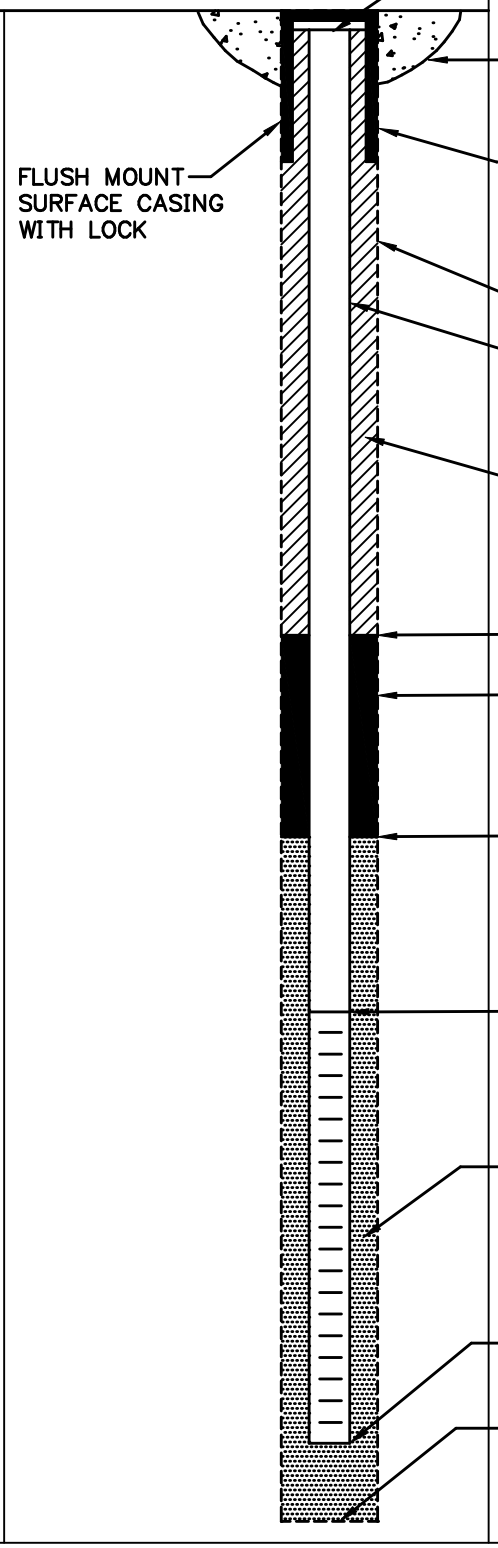
WELL NO.: _____

Tetra Tech NUS, Inc.

OVERBURDEN
MONITORING WELL SHEET
FLUSH - MOUNT

PROJECT _____	LOCATION _____	DRILLER _____
PROJECT NO. _____	BORING _____	DRILLING METHOD _____
DATE BEGUN _____	DATE COMPLETED _____	
FIELD GEOLOGIST _____		DEVELOPMENT METHOD _____
GROUND ELEVATION _____	DATUM _____	



ELEVATION TOP OF RISER: _____

TYPE OF SURFACE SEAL: _____

_____

TYPE OF PROTECTIVE CASING: _____

_____

I.D. OF PROTECTIVE CASING: _____

DIAMETER OF HOLE: _____

TYPE OF RISER PIPE: _____

_____

RISER PIPE I.D.: _____

TYPE OF BACKFILL/SEAL: _____

_____

ELEVATION/DEPTH TOP OF SEAL: _____ / _____

TYPE OF SEAL: _____

_____

ELEVATION/DEPTH TOP OF SAND: _____ / _____

ELEVATION/DEPTH TOP OF SCREEN: _____ / _____

TYPE OF SCREEN: _____

SLOT SIZE x LENGTH: _____

TYPE OF SAND PACK: _____

_____

DIAMETER OF HOLE IN BEDROCK: _____

ELEVATION / DEPTH BOTTOM OF SCREEN: _____ / _____

ELEVATION / DEPTH BOTTOM OF SAND: _____ / _____

ELEVATION/DEPTH BOTTOM OF HOLE: _____ / _____

BACKFILL MATERIAL BELOW SAND: _____

_____

ACAD: FORM_MWFM.dwg 07/20/99 INL

Tetra Tech NUS, Inc.

MONITORING WELL DEVELOPMENT RECORD

Site: _____ Depth to Bottom (ft.): _____ Project Name: _____
Well: _____ Static Water Level Before (ft.): _____ Project Number: _____
Date Installed: _____ Static Water Level After (ft.): _____ Site Geologist: _____
Date Developed: _____ Screen Length (ft.): _____ Drilling Co.: _____
Dev. Method: _____ Specific Capacity: _____
Pump Type: _____ Casing ID (in.): _____

Time	Estimated Sediment Thickness (Ft.)	Cumulative Water Volume (Gal.)	Water Level Readings (Ft. below TOC)	Temperature (Degrees C)	pH	Specific Conductance (Units _____)	Turbidity (NTU)	Remarks (odor, color, etc.)

LOW FLOW PURGE DATA SHEET

PROJECT SITE NAME: _____ WELL ID.: _____

PROJECT NUMBER: _____ DATE: _____

Time (Hrs.)	Water Level (Ft. below TOC)	Flow (mL/Min.)	pH (S.U.)	S. Cond. (mS/cm)	Turb. (NTU)	DO (mg/L)	Temp. (Celcius)	ORP mV	Salinity % or ppt	Comments

SIGNATURE(S): _____ PAGE___OF___

Tetra Tech NUS, Inc.

GROUNDWATER LEVEL MEASUREMENT SHEET

Project Name: _____ **Project No.:** _____

Location: _____ **Personnel:** _____

Weather Conditions: _____ **Measuring Device:** _____

Tidally Influenced: Yes ____ No ____ **Remarks:** _____

Well or Piezometer Number	Date	Time	Elevation of Reference Point (feet)*	Total Well Depth (feet)*	Water Level Indicator Reading (feet)*	Thickness of Free Product (feet)*	Groundwater Elevation (feet)*	Comments

* All measurements to the nearest 0.01 foot

Note: Analyte, method, and/or equipment may be deleted from form if not being performed.

FIELD ANALYTICAL LOG SHEET
GEOCHEMICAL PARAMETERS

Tetra Tech NUS, Inc.

Project Site Name: _____ Sample ID No.: _____

Project No.: _____ Sample Location: _____

Sampled By: _____ Duplicate: ☐

Field Analyst: _____ Blank: ☐

Field Form Checked as per QA/QC Checklist (initials): _____

SAMPLING DATA:

Date: / Time:	Color (Visual)	pH (S.U.)	S.C. (mS/cm)	Temp. (°C)	Turbidity (NTU)	DO (mg/l)	Salinity (%)	ORP (Eh) (+/- mv)
Method:								

SAMPLE COLLECTION/ANALYSIS INFORMATION:

ORP (Eh) (+/- mv)

Electrode Make & Model:_____

Reference Electrode (circle one): Silver-Silver Chloride / Calomel / Hydrogen

Dissolved Oxygen:

Equipment: Chemetrics Test Kit

Concentration: _____ ppm

Range Used:	Range	Method	Concentration ppm
☐	0 to 1 ppm	K-7510	
☐	1 to 12 ppm	K-7512	

Analysis Time: _____

Equipment: HACH Digital Titrator OX-DT

Analysis Time:_____

Range Used:	Range	Sample Vol.	Cartridge	Multiplier
☐	1-5 mg/L	200 ml	0.200 N	0.01
☐	2-10 mg/L	100 ml	0.200 N	0.02

Titration Count	Multiplier	Concentration
_____	x 0.01 =	mg/L
_____	x 0.02 =	mg/L

Notes:

Carbon Dioxide:

Equipment: Chemetrics Test Kit

Concentration: _____ ppm

Range Used:	Range	Method	Concentration ppm
☐	10 to 100 ppm	K-1910	
☐	100 to 1000 ppm	K-1920	
☐	250 to 2500 ppm	K-1925	

Analysis Time: _____

Equipment: HACH Digital Titrator CA-DT

Range Used:	Range	Sample Vol.	Cartridge	Multiplier
☐	10-50 mg/L	200 ml	0.3636 N	0.1
☐	20-100 mg/L	100 ml	0.3636 N	0.2
☐	100-400 mg/L	200 ml	3.636 N	1.0
☐	200-1000 mg/L	100 ml	3.636 N	2.0

Titration Count		Concentration
_____	x 0.1 =	mg/L
_____	x 0.2 =	mg/L
_____	x 1.0 =	mg/L
_____	x 2.0 =	mg/L

Standard Additions: ☐ Titrant Molarity:_____ Digits Required: 1st.:_____ 2nd.:_____ 3rd.:_____

Notes:

Hydrogen, dissolved

Equipment: Bubble strip sampling field method

Start stripper at _____ (time)

End stripper at _____ (time)

Total stripper time _____

Pump rate _____ milliliters/minute

Note: Analyte, method, and/or equipment may be deleted from form if not being performed.

FIELD ANALYTICAL LOG SHEET
GEOCHEMICAL PARAMETERS

Tetra Tech NUS, Inc.

Project Site Name: _____ Sample ID No.: _____

Project No.: _____ Sample Location: _____

Sampled By: _____ Duplicate: ☐

Field Analyst: _____ Blank: ☐

Alkalinity:

Equipment: Chemetrics Test Kit Concentration: _____ ppm

Range Used:	Range	Method	Concentration ppm
☐	10 to 100 ppm	K-9810	
☐	50 to 500 ppm	K-9815	
☐	100 to 1000 ppm	K-9820	

Analysis Time: _____

Filtered: ☐

Equipment: HACH Digital Titrator AL-DT

Range Used:	Range	Sample Vol.	Cartridge	Multiplier	Titration Count	Multiplier	Concentration
☐	10-40 mg/L	100 ml	0.1600 N	0.1	_____ & _____	x 0.1 =	mg/L
☐	40-160 mg/L	25 ml	0.1600 N	0.4	_____ & _____	x 0.4 =	mg/L
☐	100-400 mg/L	100 ml	1.600 N	1.0	_____ & _____	x 1.0 =	mg/L
☐	200-800 mg/L	50 ml	1.600 N	2.0	_____ & _____	x 2.0 =	mg/L
☐	500-2000 mg/L	20 ml	1.600 N	5.0	_____ & _____	x 5.0 =	mg/L
☐	1000-4000 mg/L	10 ml	1.600 N	10.0	_____ & _____	x 10.0 =	mg/L

Parameter:	Hydroxide	Carbonate	Bicarbonate
Relationship:			

Standard Additions: ☐ Titrant Molarity:_____ Digits Required: 1st.:_____ 2nd.:_____ 3rd.:_____

Notes:

Ferrous Iron (Fe^{2+}):

Equipment: DR-850 DR-8 _ _ Range: 0 - 3.00 mg/L Concentration: _____ ppm

Program/Module: 500nm 33

Analysis Time: _____

Equipment: IR-18C Color Wheel Range: 0 - 10 mg/L

Notes:

Filtered: ☐

Hydrogen Sulfide (H_2S):

Range: 0 - 5 mg/L

Equipment: HS-C Other: _____ Concentration: _____ ppm

Exceeded 5.0 mg/L range on color chart: ☐ Analysis Time: _____

Notes:

Sulfide (S^{2-}):

Equipment: Chemetrics Test Kit Range: 0 - 10 mg/L Concentration: _____ ppm

Range Used:	Range	Method	Concentration ppm
☐	0 to 1 ppm	K-9510	
☐	1 to 10 ppm	K-9510	

Analysis Time: _____

Filtered: ☐

Equipment: DR-850 DR-8 _ _ Range: 0 - 0.70 mg/L

Program/Module: 610nm 93

Notes:

Tetra Tech NUS, Inc.

BORING LOG

Page ___ of ___

PROJECT NAME: _____ BORING NUMBER: _____
PROJECT NUMBER: _____ DATE: _____
DRILLING COMPANY: _____ GEOLOGIST: _____
DRILLING RIG: _____ DRILLER: _____

Sample No. and Type or RQD	Depth (Ft.) or Run No.	Blows / 6" or RQD (%)	Sample Recovery / Sample Length	Lithology Change (Depth/Ft.) or Screened Interval	MATERIAL DESCRIPTION: Soil Density/ Consistency or Rock Hardness	MATERIAL DESCRIPTION: Color	MATERIAL DESCRIPTION: Material Classification	USCS *	Remarks	PID/FID Reading (ppm): Sample	Sampler BZ	Borehole**	Driller BZ**
													0

* When rock coring, enter rock brokeness.

** Include monitor reading in 6 foot intervals @ borehole. Increase reading frequency if elevated reponse read.

Drilling Area

Remarks: _____ Background (ppm): _____

Converted to Well: Yes _____ No _____ Well I.D. #: _____

Note: Analyte, method, and/or equipment may be deleted from form if not being performed.

FIELD ANALYTICAL LOG SHEET
GEOCHEMICAL PARAMETERS

Tetra Tech NUS, Inc.

Project Site Name: _____ Sample ID No.: _____

Project No.: _____ Sample Location: _____

Sampled By: _____ Duplicate: ☐

Field Analyst: _____ Blank: ☐

Sulfate (SO_4^{2-}):

Equipment: DR-850 DR-8 _ _ Range: 0 - 70 mg/L Concentration: _____ ppm

Program/Module: _____ 91 Analysis Time: _____

Standard Solution: ☐ Results: _____ Filtered: ☐

Standard Additions: ☐ Digits Required: 0.1ml:_____ 0.2ml:_____ 0.3ml:_____

Notes:

Nitrate (NO_3^--N):

Equipment: DR-850 DR-8 _ _ Range: 0 - 0.50 mg/L (1) Concentration: _____ ppm

Program/Module: 55 Analysis Time: _____ Filtered: ☐

Standard Solution: ☐ Results: _____ Nitrite Interference Treatment: ☐ Reagent Blank Correction: ☐

Standard Additions: ☐ Digits Required: 0.1ml:_____ 0.2ml:_____ 0.3ml:_____

Alternate forms: NO_2 _____ $NaNO_2$ _____ mg/L

Notes (1): If results are over limit use dilution method at step 3, 5ml sample 10ml DI result X3, range upto 1.5mg/L

Notes:

Nitrite (NO_2^--N): Concentration: _____ ppm

Equipment: DR-850 DR-8 _ _ Range: 0 - 0.350 mg/L Analysis Time: _____ Filtered: ☐

Program/Module: 62

Standard Solution: ☐ Results: _____ Reagent Blank Correction: ☐

Notes:

Manganese (Mn^{2+}): Concentration: _____ ppm

Equipment: DR-850 DR-8 _ _ Range: 0 - 20.0 mg/L Analysis Time: _____ Filtered: ☐

Program/Module: 525nm 41

Standard Solution: ☐ Results: _____ Digestion: ☐ Reagent Blank Correction: ☐

Standard Additions: ☐ Digits Required: 0.1ml:_____ 0.2ml:_____ 0.3ml:_____

Equipment: HACH MN-5 Range: 0 - 3 mg/L

Notes:

QA/QC Checklist:

All data fields have been completed as necessary: ☐

Correct measurement units are cited in the SAMPLING DATA block: ☐

Values cited in the SAMPLING DATA block are consistent with the Groundwater Sample Log Sheet: ☐

Mulitplication is correct for each *Multiplier* table: ☐

Final calulated concentration is within the appropriate *Range Used* block: ☐

Alkalinity *Relationship* is determined appropriatly as per manufacturer (HACH) instructions: ☐

QA/QC sample (e.g., Std. Additions, etc.) frequency is appropriate as per the project planning documents: ☐

Nitrite Interference treatment was used for Nitrate test if Nitrite was detected: ☐

Title block on each page of form is initialized by person who performed this QA/QC Checklist: ☐

TETRA TECH NUS
FIELD TASK MODIFICATION REQUEST FORM

Project/Installation Name	CTO & Project Number	Task Mod. Number
Modification To (e.g. Work Plan)	Site/Sample Location	Date

Activity Description: _____

Reason for Change: _____

Recommended Disposition: _____

Field Operations Leader (Signature) _____ Date _____

Approved Disposition:

Project/Task Order Manager (Signature) _____ Date _____

Distribution:

Program/Project File –
Project/Task Order Manager –
Field Operations Leader –

Other: _____

Tetra Tech NUS, Inc.

SOIL & SEDIMENT SAMPLE LOG SHEET

Project Site Name: _____
Project No.: _____

[] Surface Soil
[] Subsurface Soil
[] Sediment
[] Other: _____
[] QA Sample Type: _____

Sample ID No.: _____
Sample Location: _____
Sampled By: _____
C.O.C. No.: _____

Type of Sample:
[] Low Concentration
[] High Concentration

GRAB SAMPLE DATA:

Date: / Time: / Method: / Monitor Reading (ppm):	Depth Interval	Color	Description (Sand, Silt, Clay, Moisture, etc.)

COMPOSITE SAMPLE DATA:

Date:	Time	Depth Interval	Color	Description (Sand, Silt, Clay, Moisture, etc.)
Method:				
Monitor Readings (Range in ppm):				

SAMPLE COLLECTION INFORMATION:

Analysis	Container Requirements	Collected	Other

OBSERVATIONS / NOTES:

MAP:

Circle if Applicable:

MS/MSD	Duplicate ID No.:

Signature(s):

Tetra Tech NUS, Inc.

PROJECT: _____ **JOB #:** _____
LOCATION: _____ **DATE:** _____
PROJECT MANAGER: _____ **FOL:** _____

DAILY ACTIVITIES CHECKLIST

Startup Checklist

Activity	Yes	No	N/A
Pertinent site activities/information entered into site logbook			
All onsite personnel listed in logbook			
Required medical information onsite for all workers (TtNUS and Subcontractors)			
Required MSDS's onsite			
Proper equipment calibrations performed (list equipment)			
1 _____			
2 _____			
3 _____			
4 _____			
Calibration logs filled out			
Tailgate H&S meeting held prior to beginning field activities			
Required work permits filled out/signed			
Required utility clearances obtained			
Required PPE onsite and in use			
Information required to be posted is in place (OSHA poster, hospital route, key phone numbers, etc.)			

Exit Checklist

Activity	Yes	No	N/A
Logbooks completely and comprehensively filled out			
Field forms complete and accounted for/properly filed			
Samples properly packaged/shipped			
COCs faxed to appropriate in-house personnel			
All equipment accounted for, on charge if needed, and properly secured			
All personnel accounted for			
Arrangements made for upcoming work (permits, clearances, equipment, etc.)			
Site properly secured			

Note - not all items listed apply to every job, and some additional requirements may apply on a job-specific basis.

STANDARD OPERATING PROCEDURE
SOP-01

GLOBAL POSITIONING SYSTEM

1.0 PURPOSE

The purpose of this Standard Operating Procedure (SOP) is to provide the Field Technicians with basic instructions for operating a handheld Global Positioning System (GPS) unit allowing them to set GPS parameters in the receiver, record GPS positions on the field device, and update existing Geographic Information System (GIS) data. This SOP is specific to GIS quality data collection for Trimble-specific hardware and software.

If possible, the Trimble GeoXM or GeoXH Operators Manual should be downloaded onto the operator's personal computer for reference before or while in the field. The manual can be downloaded at http://trl.trimble.com/docushare/dsweb/Get/Document-311749/TerraSyncReferenceManual.pdf

Unless the operator is proficient in the setup and operation of the GPS unit, the Project Manager (or designee) should have the GPS unit shipped to the project-specific contact listed below in the Pittsburgh, Pennsylvania office at least five working days prior to field mobilization so project-specific shape files, data points, background images, and correct coordinate systems can be uploaded into the unit.

Tetra Tech NUS, Inc.
Attn: John Wright
661 Anderson Drive, Bldg #7
Pittsburgh, PA 15220

2.0 REQUIRED EQUIPMENT

The following hardware and software should be utilized for locating and establishing GPS points in the field:

2.1 Required GPS Hardware

- Hand-held GPS Unit capable of sub-meter accuracy (i.e. Trimble GeoXM or Trimble GeoXH). This includes the docking cradle, a/c adapter, stylus, and USB cable for data transfer.

Optional Accessories:

    - External antenna
    - Range pole
    - Hardware clamp (for mounting Geo to range pole)
    - GeoBeacon
- Indelible marker
- Non-metallic pin flags for temporary marking of positions

2.2 Required GPS Software

The following software is required to transfer data from the handheld GPS unit to a personal computer:

- Trimble TerraSync version 2.6 or later (pre-loaded onto GPS unit from vendor)

- Microsoft ActiveSync version 4.2 or later. Download to personal computer from: http://www.microsoft.com/windowsmobile/en-us/downloads/eulas/eula_activesync45_1033.mspx?ProductID=76

- Trimble Data Transfer Utility (freeware version 2.1 or later). Download to personal computer from: http://www.trimble.com/datatransfer.shtml

3.0 START-UP PROCEDURES

Prior to utilizing the GPS in the field, ensure the unit is fully charged. The unit may come charged from the vendor, but an overnight charge is recommended prior to fieldwork.

The Geo-series GPS units require a docking cradle for both charging and data transfer. The Geo-series GPS unit is docked in the cradle by first inserting the far domed end in the top of the cradled, then gently seating the contact end into the latch. The power charger is then connected to the cradle at the back end using the twist-lock connector. Attach a USB cable as needed between the cradle (B end) and the laptop/PC (A end).

It is recommended that the user also be familiar and check various Windows Mobile settings. One critical setting is the Power Options. The backlight should be set as needed to conserve power when not in use.

Start Up:

1) Power on the GPS unit by pushing the small green button located on the lower right front of the unit.

2) Utilizing the stylus that came with the GPS unit, launch **TerraSync** from the Windows Operating System by tapping on the start icon located in the upper left hand corner of the screen and then tap on **TerraSync** from the drop-down list.

3) If the unit does not default to the Setup screen, tap the Main Menu (uppermost left tab, just below the Windows icon) and select Setup.

4) If the unit was previously shipped to the Pittsburgh office for setup, you can skip directly to Section 4.0. However, to confirm or change settings, continue on to Section 3.1.

3.1 Confirm Setup Settings

Use the Setup section to confirm the TerraSync software settings. To open the Setup section, tap the Main Menu and select Setup.

1) Coordinate System
   a. Tap on the Coordinate System.
   b. Verify the project specs are correct for your specific project by scrolling through the various settings. Edit as needed and then tap OK; otherwise, tap Cancel to return to Setup Menu. **Note:** It is always best to utilize the Cancel tab rather than the OK tab if no changes are made since configurations are easily changed by mistake.
   c. Tap on the Units.
   d. Verify the user preferences are correct for your specific project by scrolling through the various settings. Edit as needed and then tap OK; otherwise, tap Cancel to return to Setup Menu.
   e. Tap Real-time Settings.
   f. Verify the Real-time Settings are correct for your specific project by scrolling through the various settings. Edit as needed and then tap OK; otherwise, tap Cancel to return to Setup Menu.
   g. The GPS unit is now configured correctly for your specific project.

4.0 ANTENNA CONNECTION

1) If a connection has been properly made with the internal antenna, a satellite icon along with the number of usable satellites will appear at the top of the screen next to the battery icon. If no connection is made (e.g.: no satellite icon), tap on the GPS tab to connect antenna.
2) At this point the GPS unit is ready to begin collecting data.

5.0 COLLECTING NEW DATA IN THE FIELD

1) From the Main Menu select Data.

2) From the Sub Menu (located below the Data tab) select New which will bring up the New Data File menu.

3) An auto-generated filename appears and should be edited for your specific project. If the integral keyboard does not appear, tap the small keyboard icon at the bottom of the screen.

4) After entering the file name, tap Create to create the new file.

5) Confirm antenna height if screen appears. Antenna height is the height that the GPS unit will be held from the ground surface (Typically 3 to 4 feet).

6) The Choose Feature screen appears.

5.1 Collecting Features

1) If not already open, the Collect Feature screen can be opened by tapping the Main Menu and selecting Data. The Sub Menu should default to Collect.

2) **Do not begin the data logging process until you are at the specific location for which you intend to log the data.**

3) A known reference or two should be shot at the beginning and at the end of each day in which the GPS unit is being used. This allows for greater accuracy during post-processing of the data.

4) Upon arriving at the specific location, tap on Point_generic as the Feature Name.

5) Tap Create to begin data logging.

6) In the Comment Box enter sample ID or location-specific information.

7) Data logging can be confirmed by viewing the writing pencil icon in the upper part of the screen. Also, the logging counter will begin. As a Rule of Thumb, accumulate a minimum of 20 readings on the counter, per point, as indicated by the logging counter before saving the GPS data.

8) Once the counter has reached a minimum number of counts (i.e. 20), tap on OK to save the data point to the GPS unit. Confirm the feature. All data points are automatically saved within the GPS unit.

9) Repeat steps 2 through 8, giving each data point a unique name or number.

Note: If the small satellite icon or the pencil icon is blinking, this is an indication the GPS unit is not collecting data. A possible problem may be too few satellites. While still in data collection mode, tap on Main Menu in upper left hand corner of the screen and select Status. Skyplot will display as the default showing the number of available satellites. To increase productivity (number of usable satellites) use the stylus to move the pointer on the productivity and precision line to the left. This will decrease precision, but increase productivity. The precision and productivity of the GPS unit can be adjusted as the number of usable satellites changes throughout the day. To determine if GPS is correctly recording data, see Section 5.2.

5.2 Viewing Data or Entering Additional Data Points to the Current File

1) To view the stored data points in the current file, tap on the Main Menu and select Map. Stored data points for that particular file will appear. Use the +/- and <-/-> icons in lower left hand corner of screen to zoom in/out and to manipulate current view.

2) To return to data collection, tap on the Main Menu and select Data. You are now ready to continue to collect additional data points.

5.3 Viewing Data or Entering Data Points from an Existing File

1) To view data points from a previous file, tap on Main Menu and select Data, then select File Manager from the Sub Menu.

4) Highlight the file you want to view and select Map from the Main Menu.

5) To add data points to this file, tap on Main Menu and select Data. Continue to collect additional data points.

6.0 NAVIGATION

This section provides instructions on navigating to saved data points in an existing file within the GPS unit.

1) From the Main Menu select Map.
2) Using the Select tool, pick the point on the map to where you want to navigate.
3) The location you select will have a box placed around the point.
4) From the Options menu, choose the Set Nav Target (aka set navigation target).
5) The location will now have double blue flags indicating this point is you navigation target.
6) From the Main Menu select Navigation.
7) The dial and data on this page will indicate what distance and direction you need to travel to reach the desired target.
8) Follow the navigation guide until you reach the point you select.
9) Repeat as needed for any map point by going back to Step 1.

7.0 PULLING IN A BACKGROUND FILE

This section provides instructions on pulling in a pre-loaded background file. These files are helpful in visualizing your current location.

1) From the Main Menu select Map, then tap on Layers, select the background file from drop down list.

2) Select the project-specific background file from the list of available files.

3) Once the selected background file appears, the operator can manipulate the screen utilizing the +/- and <-/-> functions at the bottom of the screen.

4) In operating mode, the operator's location will show up on the background file as a floating "x".

8.0 DATA TRANSFER

This section provides instructions on how to transfer stored data on the handheld GPS unit to a personal computer. Prior to transferring data from the GPS unit to a computer, Microsoft ActiveSync and Trimble Data Transfer Utility software must be downloaded to the computer from the links provided in Section 2.2 (Required GPS Software). If a leased computer is utilized in which the operator can not download files, see the Note at the end of Section 8.0.

1) See Attachment A at the end of this SOP for instructions on how to transfer data from the GPS to a personal computer.

Note: If you are unable to properly transfer data from the GPS unit to a personal computer, the unit should be shipped to the project-specific contact listed in Section 1.0 where the data will be transferred and the GPS unit then shipped back to the vendor.

9.0 SHUTTING DOWN

This section provides instruction for properly shutting down the GPS unit.

1) When shutting down the GPS unit for the day, first click on the "X" in the upper right hand corner.
2) You will be prompted to ensure you want to exit TerraSync. Select Yes.
3) Power off the GPS unit by pushing the small green button located on the bottom face of the unit.
4) Place the GPS unit in its cradle to recharge the battery overnight. Ensure the green charge light is visible on the charging cradle.

ATTACHMENT A

How to Transfer Trimble GPS Data between Data Collector and PC
original 11/21/06 (5/1/08 update) – John Wright

Remember – Coordinate System, Datum, and Units are critical!!!

Trimble Data Collection Devices:
Standard rental systems include the Trimble ProXR/XRS backpack and the newer handheld GeoXT or GeoXH units. Some of the older backpack system may come with either a RECON "PDA-style" or a TSCe or TSC1 alpha-numeric style data collector.

The software on all of the above units should be Trimble TerraSync (v 2.53 or higher – current version is 3.20) and to the user should basically look and function similar. The newer units and software versions (which should always be requested when renting) include enhancements for data processing, real-time display functions, and other features.

Data Transfer:
Trimble provides a free transfer utility program to aid in the transfer of GIS and field data. The Data Transfer Utility is a standalone program that will run on a standard office PC or laptop.

To connect a field data collector such as a RECON, GeoXM, GeoXT, GeoXH, or ProXH, you must first have Microsoft ActiveSync installed to allow the PC and the data collector to talk to one another. A standard USB cable is also needed to connect the two devices.

A CD or USB drive is provided with the data collector for use in data transfer. If needed, these programs are also available without charge via the web at:

• **Trimble Data Transfer Utility** (v 1.38) program to download the RECON or GeoXH field data to your PC: http://www.trimble.com/datatransfer.shtml

• **ActiveSync** from Microsoft to connect the data collector to the PC. The latest version (v4.5) can be found at: http://www.microsoft.com/windowsmobile/activesync/default.mspx
(see page 2 for data transfer instructions)

To Transfer Data Collected in the Field:

• Install the Data Transfer and ActiveSync software installed on your PC
• Connect the RECON or GeoXH to your PC via an A/B USB cable (blade end and square end type "HP printer" style)
• ActiveSync should auto-detect the connection and recognize the data collector
• Make sure the data file desired is CLOSED in TerraSync prior to transfer
• Connect via ActiveSync as a guest (not a partnership)
• Run the Trimble Data Transfer Utility program on your PC
• Select "***GIS Datalogger on Windows CE***" or similar selection
• Hit the green connect icon to the right - the far right area should say "***Connected to ....***" if successful
• Select the "***Receive***" data tab (under device)
• Select "***Data***" from file types on the right
• Find the file(s) needed for data transfer. You can sort the data files by clicking on the date/time header
• Select or browse to a C-drive folder you can put this file for emailing
• When the file appears on the list, hit the "***Transfer All***"
• Go to your Outlook or other email, send a message to: John.Wright@tetratech.com (or GIS department)
• Attach the file(s) you downloaded from your C-drive. For each TerraSync data file created you should have a packet of multiple data files. All need to be sent as a group – make sure you attach all files (the number of files may vary – examples include: ssf, obx, obs, gix, giw, gis, gip, gic, dd, and car)

To Transfer GIS Data from PC to the Field Device (must be converted in Pathfinder Office):

• Obtain GIS file(s) desired from GIS Department and have converted to Trimble extension
• Contact John Wright (John.Wright@tetratech.com) if needed for file conversion and upload support
• The GIS file(s) can be quickly converted if requested and sent back to the field user in the needed "Trimble xxx.imp" extension via email – then quickly downloaded from Outlook to your PC for transfer
• Install the Data Transfer and ActiveSync software installed on your PC
• Connect the RECON or GeoXH to your PC via an A/B USB cable (blade end and square end type "HP printer" style)
• ActiveSync should auto-detect the connection and recognize the data collector
• Connect via ActiveSync as a guest (not a partnership)
• Run the Trimble Data Transfer Utility program on your PC
• Select "***GIS Datalogger on Windows CE***" or similar selection
• Hit the green connect icon to the right - the far right area should say "***Connected to ....***" if successful
• Select the "**Send**" data tab (under device)
• Select "**Data**" from file types on the right (you can also send background files)
• Browse to the location of the data on your PC (obtain the file from Pathfinder Office or from the person who converted the data for field use)
• Select the options as appropriate for the name and location of the data file to go on the data collector (usually you can choose main memory or a data storage card)
• When the file(s) appears on the list, hit the "***Transfer All***"
• Run TerraSync on the field device and open the existing data files. Your transferred file should appear (make sure you have selected Main Memory, Default, or Storage Card as appropriate)

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		Effective Date 09/03	Revision 1
		Applicability Tetra Tech NUS, Inc.	
		Prepared Risk Assessment Department	
Subject SAMPLE NOMENCLATURE		Approved D. Senovich	

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1.0 PURPOSE

The purpose of this document is to specify a consistent sample nomenclature system that will facilitate subsequent data management in a cost-effective manner. The sample nomenclature system has been devised such that the following objectives can be attained:

- Sorting of data by matrix.
- Sorting of data by depth.
- Maintenance of consistency (field, laboratory, and data base sample numbers).
- Accommodation of all project-specific requirements.
- Accommodation of laboratory sample number length constraints (maximum of 20 characters).

2.0 SCOPE

The methods described in this procedure shall be used consistently for all projects requiring electronic data.

3.0 GLOSSARY

None.

4.0 RESPONSIBILITIES

Program Manager - It shall be the responsibility of the Program Manager (or designee) to inform contract-specific Project Managers of the existence and requirements of this Standard Operating Procedure.

Project Manager - It shall be the responsibility of the Project Manager to determine the applicability of this Standard Operating Procedure based on: (1) program-specific requirements, and (2) project size and objectives. It shall be the responsibility of the Project Manager (or designee) to ensure that the sample nomenclature is thoroughly specified in the relevant project planning document (e.g., sampling and analysis plan) and is consistent with this Standard Operating Procedure if relevant. It shall be the responsibility of the project manager to ensure that the Field Operations Leader is familiar with the sample nomenclature system.

Field Operations Leader - It shall be the responsibility of the Field Operations Leader to ensure that all field technicians or sampling personnel are thoroughly familiar with this Standard Operating Procedure and the project-specific sample nomenclature system. It shall be the responsibility of the Field Operations Leader to ensure that the sample nomenclature system is used during all project-specific sampling efforts.

5.0 PROCEDURES

5.1 Introduction

The sample identification (ID) system can consist of as few as 8 but not more than 20 distinct alpha-numeric characters. The sample ID will be provided to the laboratory on the sample labels and chain-of-custody forms. The basic sample ID provided to the lab has three segments and shall be as follows where "A" indicates "alpha," and "N" indicates "numeric":

A or N 3- or 4-Characters	AAA 2- or 3-Characters	A or N 3- to 6-Characters
Site Identifier	Sample Type	Sample Location

Additional segments may be added as needed. For example:

(1) Soil and Sediment Sample ID

A or N 3- or 4-Characters	AAA 2- or 3-Characters	A or N 3- to 6-Characters	NNNN 4-Characters
Site Identifier	Sample Type	Sample Location	Sample Depth

(2) Aqueous (groundwater or surface water) Sample ID

A or N 3- or 4-Characters	AAA 2- or 3-Characters	A or N 3- to 6-Characters	NN 2-Characters	-A
Site Identifier	Sample type	Sample Location	Round Number	Filtered Sample only

(3) Biota Sample ID

A or N 3- or 4-Characters	AAA 2- or 3-Characters	A or N 3- to 6-Characters	AA 2-Characters	NNN 3-Characters
Site Identifier	Sample Type	Sample Location	Species Identifier	Sample Group Number

5.2 Sample Identification Field Requirements

The various fields in the sample ID will include but are not limited to the following:

- Site Identifier
- Sample Type
- Sample Location
- Sample Depth
- Sampling Round Number
- Filtered
- Species Identifier
- Sample Group Number

The site identifier must be a three- or four-character field (numeric characters, alpha characters, or a mixture of alpha and numeric characters may be used). A site number is necessary since many facilities/sites have multiple individual sites, SWMUs, operable units, etc. Several examples are presented in Section 5.3 of this SOP.

The sample type must be a two- or three-character alpha field. Suggested codes are provided in Section 5.3 of this SOP.

The sample location must be at least a three-character field but may have up to six-characters (alpha, numeric, or a mixture). The six-characters may be useful in identifying a monitoring well to be sampled or describing a grid location.

The sample depth field is used to note the depth below ground surface (bgs) at which a soil or sediment sample is collected. The first two numbers of the four-number code specify the top interval, and the third and fourth specify the bottom interval in feet bgs of the sample. If the sample depth is equal to or greater than 100, then only the top interval would be represented and the sampling depth would be truncated to

three-characters. The depths will be noted in whole numbers only; further detail, if needed, will be recorded on the sample log sheet, boring log, logbook, etc.

A two-digit round number will be used to track the number of aqueous samples taken from a particular aqueous sample location. The first sample collected from a location will be assigned the round identifier 01, the second 02, etc. This applies to both existing and proposed monitoring wells and surface water locations.

Aqueous samples that are field filtered (dissolved analysis) will be identified with an "-F" in the last field segment. No entry in this segment signifies an unfiltered (total) sample.

The species identifier must be a two-character alpha field. Several suggested codes are provided in Section 5.3 of this SOP.

The three digit sample group number will be used to track the number of biota sample groups (a particular group size may be determined by sample technique, media type, the number of individual caught, weight issues, time, etc.) by species and location. The first sample group of a particular species collected from a given location will be assigned the sample group number 001 and the second sample group of the same species collected from the same location will be assigned the sample group number 002.

5.3 Example Sample Field Designations

Examples of each of the fields are as follows:

Site Identifier - Examples of site numbers/designations are as follows:

A01 - Area of Concern Number 1
125 - Solid Waste Management Unit Number 125
000 - Base or Facility Wide Sample (e.g., upgradient well)
BBG - Base Background

The examples cited are only suggestions. Each Project Manager (or designee) must designate appropriate (and consistent) site designations for their individual project.

Sample Type - Examples of sample types are as follows:

AH - Ash Sample
AS - Air Sample
BM - Building Material Sample
BSB - Biota Sample Full Body
BSF - Biota Sample Fillet
CP - Composite Sample
CS - Chip Sample
DS - Drum Sample
DU - Dust Sample
FP - Free Product
IDW - Investigation Derived Waste Sample
LT - Leachate Sample
MW - Monitoring Well Groundwater Sample
OF - Outfall Sample
RW - Residential Well Sample
SB - Soil Boring Sample
SD - Sediment Sample
SC - Scrape Sample

SG - Soil Gas Sample
SL - Sludge Sample
SP - Seep Sample
SS - Surface Soil Sample
ST - Storm Sewer Water Sample
SW - Surface Water Sample
TP - Test Pit Sample
TW - Temporary Well Sample
WC - Well Construction Material Sample
WP - Wipe Sample
WS - Waste/Solid Sample
WW - Wastewater Sample

Sample Location - Examples of the location field are as follows:

001 - Monitoring Well 1
N32E92 - Grid location 32 North and 92 East
D096 - Investigation derived waste drum number 96

Species Identifier - Examples of species identifier are as follows:

BC - Blue Crab
GB - Blue Gill
CO - Corn
SB - Soybean

5.4 Examples of Sample Nomenclature

The first round monitoring well groundwater sample collected from existing monitoring well 001 at SWMU 16 for a filtered sample would be designated as 016MW00101-F.

The second round monitoring well groundwater sample collected from existing monitoring well C20P2 at Site 23 for an unfiltered sample would be designated as 023MWC20P202.

The second surface water sample collected from point 01 at SWMU 130 for an unfiltered sample would be designated as 130SW00102.

A surface soil sample collected from grid location 32 North and 92 East at Site 32 at the 0- to 2-foot interval would be designated as 032SSN32E920002.

A subsurface soil sample from soil boring 03 at SWMU 32 at an interval of 4 to 5 feet bgs would be designated as 032SB0030405.

A sediment sample collected at SWMU 19 from 0 to 6 inches at location 14 would be designated as 019SD0140001. The sample data sheet would reflect the precise depth at which this sample was collected.

During biota sampling for full body analysis the first time a minnow trap was checked at grid location A25 of SWMU 1415 three small blue gills were captured, collected and designated with the sample ID of 1415BSBA25BG001. The second time blue gill were collected at the same location (grid location A25 at SWMU 1415) the sample ID designation given was 1415BSBA25BG002.

Note: No dash (-) or spacing is used between the segments with the exception of the filtered segment. The "F" used for a filtered aqueous sample is preceded by a dash "-F".

5.5 Field Quality Assurance/Quality Control (QA/QC) Sample Nomenclature)

Field QA/QC will be designated using a different coding system. The QC code will consist of a three- to four-segment alpha-numeric code that identifies the sample QC type, the date the sample was collected, and the number of this type of QC sample collected on that date.

AA	NNNNNN	NN	-F
QC Type	Date	Sequence Number (per day)	Filtered (aqueous only, if needed)

The QC types are identified as:

TB = Trip Blank
RB = Rinsate Blank (Equipment Blank)
FD = Field Duplicate
AB = Ambient Conditions Blank
WB = Source Water Blank

The sampling time recorded on the Chain-of-Custody Form, labels, and tags for duplicate samples will be 0000 so that the samples are "blind" to the laboratory. Notes detailing the sample number, time, date, and type will be recorded on the routine sample log sheets and will document the location of the duplicate sample (sample log sheets are not provided to the laboratory). Documentation for all other QC types (TB, RB, AB, and WB) will be recorded on the QC Sample Log sheet (see SOP on Field Documentation).

5.6 Examples of Field QA/QC Sample Nomenclature

The first duplicate of the day for a filtered ground water sample collected on June 3, 2000 would be designated as FD06030001-F.

The third duplicate of the day taken of a subsurface soil sample collected on November 17, 2003 would be designated as FD11170303.

The first trip blank associated with samples collected on October 12, 2000 would be designated as TB10120001.

The only rinsate blank collected on November 17, 2001 would be designated as RB11170101.

6.0 DEVIATIONS

Any deviation from this SOP must be addressed in detail in the site specific planning documents.

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		Effective Date 01/29/01	Revision 2
		Applicability Tetra Tech NUS, Inc.	
		Prepared Management Information Systems Department	
Subject DATABASE RECORDS AND QUALITY ASSURANCE		Approved D. Senovich	

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1.0 PURPOSE

The purpose of this document is to specify a consistent procedure for the quality assurance review of electronic and hard copy databases. This SOP outlines the requirements for establishment of a Database Record File, Quality Assurance review procedures, and documentation of the Quality Assurance Review Process.

2.0 SCOPE

The methods described in this Standard Operating Procedure (SOP) shall be used consistently for all projects managed by Tetra Tech NUS (TtNUS).

3.0 GLOSSARY

Chain-of-Custody Form - A Chain-of-Custody Form is a printed form that accompanies a sample or a group of samples from the time of sample collection to the laboratory. The Chain-of-Custody Form is retained with the samples during transfer of samples from one custodian to another. The Chain-of-Custody Form is a controlled document that becomes part of the permanent project file. Chain-of-Custody and field documentation requirements are addressed in SOP SA-6.1.

Electronic Database - A database provided on a compact laser disk (CD). Such electronic databases will generally be prepared using public domain software such as DBase, RBase, Oracle, Visual FoxPro, Microsoft Access, Paradox, etc.

Hardcopy Database - A printed copy of a database prepared using the software discussed under the definition of an electronic database.

Form I - A printed copy of the analytical results for each sample.

Sample Tracking Summary - A printed record of sample information including the date the samples were collected, the number of samples collected, the sample matrix, the laboratory to which the samples were shipped, the associated analytical requirements for the samples, the date the analytical data were received from the laboratory, and the date that validation of the sample data was completed.

4.0 RESPONSIBILITIES

Database Records Custodian - It shall be the responsibility of the Database Records Custodian to update and file the Sample Tracking Summaries for all active projects on a weekly basis. It shall be the responsibility of the Database Records Custodian to ensure that the most recent copies of the Sample Tracking Summaries are placed in the Database Records file. It shall be the responsibility of the Database Records Custodian to ensure that a copy of all validation deliverables is provided to the Project Manager (for placement in the project file). It shall be the responsibility of the Database Records Custodian to ensure that photocopies of all validation deliverables and historical data and reports (as applicable) are placed in the Database Records file.

Data Validation Coordinator - It shall be the responsibility of the Data Validation Coordinator (or designee) to ensure that the Sample Tracking Summaries are maintained by the Database Records Custodian. It shall be the responsibility of the Data Validation Coordinator (or designee) to ensure that photocopies of all data validation deliverables are placed in the applicable Database Records file by the Database Records Custodian.

Earth Sciences Department Manager - It shall be the responsibility of the Earth Sciences Department Manager (or equivalent) to ensure that all field personnel are familiar with the requirements of this Standard Operating Procedure (specifically Section 5.5).

FOL - It shall be the responsibility of the FOL (FOL) of each project to ensure that all field technicians or sampling personnel are thoroughly familiar with this SOP, specifically regarding provision of the Chain-of-Custody Forms to the Database Records Custodian. Other responsibilities of the FOL are described in Sections 5.4 and 5.5.

Management Information Systems (MIS) Manager - It shall be the responsibility of the MIS Manager to ensure that copies of original electronic deliverables (CDs) are placed in both the project files and the Database Records File. It shall be the responsibility of the MIS Manager (or designee) to verify the completeness of the database (presence of all samples) in both electronic and hardcopy form in the Database Records File. It shall be the responsibility of the MIS Manager to ensure that Quality Assurance Reviews are completed and are attested to by Quality Assurance Reviewers. It shall be the responsibility of the MIS Manager to ensure that records of the Quality Assurance review process are placed in the Database Records File. It shall be the responsibility of the MIS Manager to ensure that both electronic and hardcopy forms of the final database are placed in both the project and the Database Record File. It shall be the responsibility of the MIS Manager to ensure that data validation qualifiers are entered in the database.

Furthermore, it shall be the responsibility of the MIS Manager to participate in project planning at the request of the Project Manager, specifically with respect to the generation of level of effort and schedule estimates. To support the project planning effort, the MIS Manager shall provide a copy of the MIS Request From included as Attachment A to the project manager. It shall be the responsibility of the MIS Manager to generate level of effort and budget estimates at the time database support is requested if a budget does not exist at the time of the request. The MIS Request Form shall be provided to the Project Manager at the time of any such requests. It shall be the responsibility of the MIS Manager to notify the Project Manager of any anticipated level of effort overruns or schedule noncompliances as soon as such problems arise along with full justification for any deviations from the budget estimates (provided they were generated by the MIS Manager). It shall be the responsibility of the MIS Manager to document any changes to the scope of work dictated by the Project Manager, along with an estimate of the impact of the change on the level of effort and the schedule.

Program/Department Managers - It shall be the responsibility of the Department and/or Program Managers (or designees) to inform their respective department's Project Managers of the existence and requirements of this SOP.

Project Manager - It shall be the responsibility of each Project Manager to determine the applicability of this SOP based on: (1) program-specific requirements, and (2) project size and objectives. It shall be the responsibility of the Project Manager (or designee) to ensure that the FOL is familiar with the requirements regarding Chain-of-Custody Form provision to the Database Records Custodian. It shall be the responsibility of the Project Manager (or designee) to determine which, if any, historical data are relevant and to ensure that such data (including all relevant information such as originating entity, sample locations, sampling dates, etc.) are provided to the Database Records Custodian for inclusion in the Database Records File. It shall be the responsibility of the Project Manager to obtain project planning input regarding the level of effort and schedule from the MIS Manager. It shall be the responsibility of the Project Manager to complete the database checklist (Attachment A) to support the level of effort and schedule estimate and to facilitate database preparation and subroutine execution.

Risk Assessment Department Manager - It shall be the responsibility of the Risk Assessment Department Manager to monitor compliance with this Standard Operating Procedure, to modify this SOP as necessary, and to take corrective action if necessary. Monitoring of the process shall be completed on a quarterly basis.

Quality Assurance Reviewers - It shall be the responsibility of the Quality Assurance Reviewers to verify the completeness of the sample results via review of the Chain-of-Custody Forms and Sample Tracking Summaries. It shall be the responsibility of the Quality Assurance Reviewers to ensure the correctness of the database via direct comparison of the hardcopy printout of the database and the hardcopy summaries of the original analytical data (e.g., Form Is provided in data validation deliverables). Correctness includes the presence of all relevant sample information (all sample information fields), agreement of the laboratory and database analytical results, and the presence of data validation qualifiers.

Quality Manager - It shall be the responsibility of the Quality Manager to monitor compliance with this Standard Operating Procedure via routine audits.

5.0 PROCEDURES

5.1 Introduction

Verification of the accuracy and completeness of an electronic database can only be accomplished via comparison of a hardcopy of the database with hardcopy of all relevant sample information. The primary purposes of this SOP are to ensure that 1) all necessary hardcopy information is readily available to Quality Assurance Reviewers; 2) ensure that the Quality Assurance review is completed in a consistent and comprehensive manner, and; 3) ensure that documentation of the Quality Assurance review process is maintained in the project file.

5.2 File Establishment

A Database Record file shall be established for a specific project at the discretion of the Project Manager. Initiation of the filing procedure will commence upon receipt of the first set of Chain-of-Custody documents from a FOL or sampling technician. The Database Record Custodian shall establish a project-specific file for placement in the Database Record File. Each file in the Database Record File shall consist of standard components placed in the file as the project progresses. Each file shall be clearly labeled with the project number, which shall be placed on the front of the file drawer and on each and every hanging file folder relevant to the project. The following constitute the minimum components of a completed file:

- Electronic Deliverables
- Sample Tracking Forms
- Chain-of-Custody Forms
- Data Validation Letters
- Quality Assurance Records

5.3 Electronic Deliverables

The format of electronic deliverables shall be specified in the laboratory procurement specification and shall be provided by the laboratory. The integrity of all original electronic data deliverables shall be maintained. This shall be accomplished via the generation of copies of each electronic deliverable provided by the laboratory. The original electronic deliverable shall be provided to the project manager for inclusion in the project file. A copy of the original electronic deliverable shall be placed in the Database Record File. The second copy shall be maintained by the MIS Manager (or designee) to be used as a working copy.

5.4 Sample Tracking Forms

Updated versions of the sample tracking form for each relevant project shall be maintained by the Database Record Custodian. The Sample Tracking Forms shall be updated any time additional Chain-of-Custody Forms are received from a FOL or sampling technician, or at any time that data are received from a laboratory, or at any time that validation of a given data package (sample delivery group) is completed. The Data Validation Coordinator shall inform the Database Record Custodian of the receipt of any data packages from the laboratory and of completion of validation of a given data package to facilitate updating of the Sample Tracking Form. The Database Record Custodian shall place a revised copy of the Sample Tracking Form in the Database Record File anytime it has been updated. Copies of the updated Sample Tracking Form shall also be provided to the project manager to apprise the project manager of sample package receipt, completion of validation, etc.

5.5 Chain-of-Custody Forms

The Chain-of-Custody Forms for all sampling efforts will be used as the basis for (1) updating the Sample Tracking Form, and (2) confirming that all required samples and associated analyses have been completed. It shall be the responsibility of the FOL (or sample technician) to provide a photocopy of all Chain-of-Custody Forms to the Database Record Custodian immediately upon completion of a sampling effort. The Database Record Custodian shall then place the copies of the Chain-of-Custody Form(s) in the Database Record File. Upon receipt of a sample data package from an analytical laboratory, the Data Validation Coordinator shall provide a copy of the laboratory Chain-of-Custody Form to the Database Record Custodian. The Database Record Custodian shall use this copy to update the Sample Tracking Summary and shall place the copy of the laboratory-provided Chain-of-Custody Form in the Database Record File. The photocopy of the laboratory-provided Chain-of Custody Form shall be stapled to the previously filed field copy. Upon receipt of all analytical data, two copies of the Chain-of-Custody will therefore be in the file. Review of the Chain-of-Custody Forms will therefore be a simple mechanism to determine if all data have been received. Chain-of-Custody is addressed in SOP SA-6.1.

5.6 Data Validation Letters

All data validation deliverables (or raw data summaries if validation is not conducted) shall be provided for inclusion in both the Database Record File and the project file. If USEPA regional- or client-specific requirements are such that Form Is (or similar analytical results) need not be provided with the validation deliverable, copies of such results must be appended to the deliverable. It is preferable, although not essential that the validation qualifiers be hand-written directly on the data summary forms. The data validation deliverables (and attendant analytical summaries) will provide the basis for direct comparison of the database printout and the raw data and qualifiers.

5.7 Historical Data

At the direction of the Project Manager, historical data may also be included in a project-specific analytical database. In the event that historical data are germane to the project, hardcopy of the historical data must be included in the Database Record File. Historical data may be maintained in the form of final reports or as raw data. The information contained in the historical data file must be sufficient to identify its origin, its collection date, the sample location, the matrix, and any and all other pertinent information. All available analytical data, Chain-of-Custody Forms, boring logs, well construction logs, sample location maps, shall be photocopied by the Project Manager (or designee) and placed in one or more 3-ring binders. All information shall be organized chronologically by matrix. It shall be the responsibility of the Project Manager (or designee) to ensure that all inconsistencies between analytical data, Chain-of-Custody Forms, boring logs, sample log sheets, and field logbooks are identified and corrected. The Project Manager (or designee) shall decide which nomenclature is appropriate and edit, initial and date all relevant forms. Data entry may only be performed on information that has undergone the aforementioned

editing process, thereby having a direct correlation between hardcopy information and what will become the electronic database.

6.0 RECORDS

Records regarding database preparation and quality assurance review include all those identified in the previous section. Upon completion of the database task, records from the file will be forwarded to the Project Manager for inclusion in the project file, or will be placed in bankers boxes (or equivalent) for storage. The final records for storage shall include the following minimum information on placards placed on both the top and end of the storage box:

Database Record File
PROJECT NUMBER: ____
SITE NAME: _____
DATE FILED: __/__/__
SUMMARY OF CONTENTS ENCLOSED
BOX _ OF _

Project- or program-specific record keeping requirements shall take precedence over the record keeping requirements of this SOP.

ATTACHMENT A

MIS REQUEST FORM

Tetra Tech NUS, Inc.

Project Name: | Request Date:
CTO: | Date Data Available for Production:
Project Manager: | Request in Support of:
Requestor: | Database Lead:
Program/Client: | GIS Lead:
State/EPA Region: | Statistics Lead:
| Risk Lead:
Site Name(s) (Area, OU, etc.):
Sampling Date(s):
Matrix: ☐ GW ☐ SO ☐ SD ☐ SW ☐ Other:

Labels: ☐ Labels needed for an upcoming sampling event _____ Total # of Samples
_____ Estimated Hours
_____ Due Date
_____ Complete ETS Charge No.
_____ FOL
Additional Instructions:

Data Entry:
☐ Chemical data needs to be entered from hardcopy _____ Estimated # of Samples
☐ Chemical data needs to be formated electronically
☐ Field analytical data needs to be entered from hardcopy
☐ Geologic data needs to be entered from hardcopy
☐ Hydrology data needs to be entered from hardcopy
_____ Estimated Hours
_____ Due Date
_____ Complete ETS Charge No.
Additional Instructions:

Tables:
☐ Full Data Printout
☐ Summary of Positive Hits
☐ Occurance and Distribution ☐ with criteria
☐ Sampling Analytical Summary
☐ Other:
_____ Estimated Hours
_____ Due Date
_____ Complete ETS Charge No.
Additional Instructions:

GIS:
☐ General Facility Location
☐ Site Location
☐ Potentiometric Contours/Groundwater Flow
☐ Sample Location Proposed
☐ Sample Location Existing
☐ Tag Map Single Round
☐ Tag Map Multiple Round
☐ Isoconcentrations
☐ Chart Map
☐ 3D Visualization
☐ EGIS CD
☐ Other:
_____ Estimated Hours
_____ Due Date
_____ Complete ETS Charge No.
Additional Instructions:

Statistics: ☐ Yes
_____ Estimated Hours
_____ Due Date
_____ Complete ETS Charge No.
Additional Instructions:

Geostatistics: ☐ Yes
_____ Estimated Hours
_____ Due Date
_____ Complete ETS Charge No.
Additional Instructions:

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		Effective Date 06/99	Revision 1
		Applicability Tetra Tech NUS, Inc.	
		Prepared Earth Sciences Department	
Subject SITE RECONNAISSANCE		Approved D. Senovich	

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1.0 PURPOSE

The purpose of a site reconnaissance is to collect both general and technical information which will support the scoping, scheduling, implementing project activities, and writing reports for an environmental investigation. This procedure is not intended as a guide for Phase I investigations or for Environmental Baseline Survey activities.

2.0 SCOPE

This procedure is applicable to the performance of a site reconnaissance for initial site characterization. The steps necessary to develop and carry out a site reconnaissance are presented here. These steps include a list of equipment and items which may be needed, areas of special interest during field observations, and methods by which the field observation team can ensure that necessary and appropriate observations have been made.

3.0 GLOSSARY

Site reconnaissance. An onsite inspection program used to identify site-specific conditions that control scheduling, manpower, and affect costs. A site reconnaissance usually consists of visual observations and, often, the use of field monitoring instruments to identify potential health and safety threats and potential sampling locations for site evaluation during subsequent field investigations.

4.0 RESPONSIBILITIES

Field Operations Leader (FOL) is responsible for ensuring that the survey is carried out in sufficient detail. To accomplish this, the FOL must assign the proper personnel and equipment to characterize the site adequately, in accordance with the requirements defined in this procedure and best engineering practices. Other disciplines which may be applicable include (but are not limited to): Geology/Hydrogeology; Health and Safety; Ecological Specialists; and/or Engineering. In addition, the FOL is responsible for supervising equipment preparation, including necessary calibrations, and supervising field data collection and documentation in accordance with the methods described in all referenced standard operation procedures.

Project Manager is responsible for the following:

- Supervising the retrieval and examination of available, applicable information regarding the site.
- Obtaining appropriate program approvals and ensuring the preparation of a site Health and Safety plan for the site reconnaissance.
- Coordinating the field activities with the client and regulatory agencies, as applicable.

Field Personnel are primarily responsible for observing and documenting, either through written documentation or photographic evidence, the site reconnaissance. Field personnel will take direction from the FOL.

5.0 PROCEDURES

5.1 Equipment Items/Needed

Below is a list of items that may be useful when conducting a site reconnaissance. All, or a portion of these items may be required, depending upon the objective of the site reconnaissance.

- Health and Safety equipment and information as required by the Site Safety Officer.
- Maps (U.S.G.S. quadrangle, geologic maps, street and highway maps, and client facility maps).
- Geologic tools (compass, tape measure, hand level, camera, etc.).
- Physical monitoring equipment, if applicable (PID, Immunoassay Test Kits, etc.)
- Regional publications (U.S.G.S reports, water well surveys, U.S.D.A. soil conservation surveys, etc.).
- Site-specific publications by previous investigators (EPA aerial photographic analyses, remedial investigation reports, data on waste disposal practices, boring logs, etc.).
- Marking items (ink markers, surveyor's flagging, spray paint, pin flags, wooden stakes).
- Field notebooks.
- Local telephone book with yellow pages (for obtaining utilities, site trailer, living accommodations, etc.).

Sufficient time will be required in order to obtain some of the aforementioned material. In general, most publications can be obtained in time to be used in the site reconnaissance if ordered approximately 2 weeks before the actual site visit takes place.

5.2 Observations

A site reconnaissance usually requires one to two days, however, additional time may be needed depending upon the objective, site size, etc. The following observations, when applicable, should be documented either on a site map, field notebook, or photographed.

- General Site Access. It should be noted whether site roads provide access to all proposed work locations, or if it will be necessary to prepare access roads with either a backhoe, dozer, chain saws, etc., in order to get drill rigs, excavators, or other work vehicles to specific locations. If temporary driveways must be constructed from existing public roads, regulatory permits may be required. Military facilities may have specific security requirements which require detailed clearance procedures.
- Location of the Command Post or Site Trailer and Sanitary Facilities. The ideal location for the site trailer and sanitary facilities is a level area, within an uncontaminated zone, and centralized in order to provide easy access to work areas on the site. However, certain utility companies may require that the site trailer be placed within a specified radius (usually 100 feet), of the nearest utility pole. Contact the necessary utility companies and inquire about the requirements regarding service before conducting the site reconnaissance. Information that may be required by the utility companies is: type of electric service needed (inquire with trailer vendor for this information); and utility pole number of interest (pole numbers are usually stamped on a brass plate on the pole).

- Potable Water Sources. Local fire departments may allow access to fire hydrants. Private water delivery companies may also be available in the area.

- Sources of Possible Contamination. Drums, tanks, sludge areas, areas of stressed vegetation, fill areas, and leachate seeps may indicate where sources of contamination exist. Filler pipes protruding from the ground surface may indicate the presence of underground storage tanks. Areas where the original ground surface has been reworked may be contaminated fill areas that have since been buried and covered with natural material. Previous environmental investigations may also identify source areas.

- Location of Decon Areas and Storage/Disposal Areas for Equipment and Wastes Generated by Field Activities.

- Locations of Surface Water Bodies. The locations of surface water bodies, both man-made and natural, and their relation to topographic highs may give an indication of the groundwater flow direction in the area (groundwater flow typically follows topography with the topographic highs serving as groundwater recharge areas, and the surface waters at topographic lows serve as groundwater discharge areas). Visible signs of contamination, the existence of aquatic life, flow rates, and approximate levels should also be observed and noted. Check if the surface water bodies could potentially be impacted by field activities. If so, appropriate sedimentation and erosion controls will be required.

- Existing Wells. Existing monitoring wells, or domestic wells within the site and off site, should be noted on a map, and access checked to see if the wells can be used for data collection.

- Outcrops. Outcrops can be useful in providing hydrogeologic data (lithologic description, strike and dip information, fracture and joint system analysis, identification of moist zones, etc.) Outcrops may occur naturally or be a part of a man-made feature such as a road-cut.

- Lineaments. A lineament is a straight lengthy feature on the earth's surface which is expressed topographically as a line of depression. Stream beds, vegetation patterns or soil characteristics may be aligned or controlled by this feature. Lineaments are due in some cases to the presence of intense jointing or faults beneath the ground surface. Groundwater in the bedrock may follow lineaments. Lineaments should be noted on site maps and described in the field notebooks.

- Bench or Property Markers. Benchmarks or property markers should be marked with paint or surveyor's flagging if encountered during a site reconnaissance. Surveyors may need to use these markers as a reference point when surveying. Benchmarks are typically a brass plate secured in concrete in the ground with numbering on the top. Property markers can range from a stake driven into the ground to a rock protruding from the ground surface. Facility contacts may also be aware of local benchmarks used during the course of other environmental or public work projects.

- Metal Cultural Effects. Overhead power lines, railroad tracks, junk automobiles, fences, etc. will greatly affect certain geophysical surveys. These features should be noted while conducting a site reconnaissance.

6.0 RECORDS

The data collected during a site reconnaissance may have to be compiled into a trip report when returning from the field. This trip report can then be distributed to the project team. A site reconnaissance checklist is located in Attachment A which can be copied and used while conducting the site reconnaissance.

ATTACHMENT A

SITE RECONNAISSANCE CHECKLIST

SITE SKETCH

Include the following as appropriate:

- Site Name
- Site location
- Site Boundaries
- Entrance locations
- Access Roads and Security Requirements
- Disposal locations
- Storage areas
- Office areas
- Well locations
- Treatment facility locations
- Surface drainage, outcrops, general topography descriptions
- Cultural interferences

CHEMICAL STORAGE FACILITIES DESCRIPTION

- Storage tanks - numbers, volumes, condition, contents, etc.
- Drums - number, conditions, labeling, etc.
- Lagoons and surface pits - number, size, use of liner, contents, etc.

TREATMENT SYSTEMS

Note the presence of any treatment systems. These can be difficult to evaluate visually. One should appraise general appearance, maintenance and visual integrity; ask operators for any monitoring records; note presence of odors; and visually characterize any effluents or residues. Describe type of wastes and volumes treated.

- Incinerators
- Flocculation/filtration
- Chemical/physical treatment
- Biological treatment
- Volume reduction
- Waste recycling
- Compositing
- Other

ATTACHMENT A
SITE RECONNAISSANCE CHECKLIST
PAGE TWO

DISPOSAL FACILITIES

Note the presence and use of any of the following operations. Include a description of the size, use of liners, soil type, and the presence of leachate. Provide a description of management practices. Interview site workers if possible. Describe waste types.

- Landfills
- Land forms
- Open dump
- Surface impoundment
- Underground injection
- Incineration

Also, records for disposal of concentrated/containerized waste should be reviewed.

HAZARDOUS SUBSTANCE CHARACTERISTICS

Ask facility contacts for manifests, inventories, or monitoring reports. Note markings on containers.

- Chemical identities
- Quantities
- Hazard characteristics (toxic, explosive, flammable, etc.)
- Container markings
- Monitoring data, other analytical data
- Physical state (liquid, solid, gas, sludge)

CHEMICAL PROCESS INFORMATION

- Manufacturing processes and chemicals
- Off-specification or by-product disposal processes
- Housekeeping practices
- Locations of Plant Operations

ATTACHMENT A
SITE RECONNAISSANCE CHECKLIST
PAGE THREE

HYDROGEOLOGIC ASSESSMENT

Look for situations that promote hazardous substance migration, i.e., porous soils, fractured bedrock formations, shallow water table and karst features.

- Soil type
- Surface water features
- Surface drainage pattern
- Outcrop studies
- Water wells (use, water depth, and construction details)
- Erosion potential
- Flooding potential
- Climatology

IDENTIFICATION OF SENSITIVE RECEPTORS

- Number and locations of private homes
- Public buildings including tenant usage
- Areas of dead or dying vegetation or animals
- Presence of sensitive ecosystems (wetlands, tidal marshes, etc.)
- Other public use areas (roads, parks, etc.)
- Natural areas

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		Effective Date 09/03	Revision 3
		Applicability Tetra Tech NUS, Inc.	
		Prepared Earth Sciences Department	
Subject DIRECT PUSH TECHNOLOGY (GEOPROBE®/HYDROPUNCH™)		Approved D. Senovich	

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1.0 PURPOSE

The purpose of this procedure is to provide general reference information on Direct Push Technology (DPT). DPT is designed to collect soil, groundwater, and soil gas samples without using conventional drilling techniques. The advantage of using DPT over conventional drilling includes the generation of little or no drill cuttings, sampling in locations with difficult accessibility, reduced overhead clearance requirements, no fluid introduction during probing, and typical lower costs per sample than with conventional techniques. Disadvantages include a maximum penetration depth of approximately 15 to 40 feet in dense soils (although it may be as much as 60 to 80 feet in certain types of geological environments), reduced capability of obtaining accurate water-level measurements, and the inability to install permanent groundwater monitoring wells. The methods and equipment described herein are for collection of surface and subsurface soil samples and groundwater samples. Soil gas sampling is discussed in SOP SA-2.4.

2.0 SCOPE

This procedure provides information on proper sampling equipment and techniques for DPT. Review of the information contained herein will facilitate planning of the field sampling effort by describing standard sampling techniques. The techniques described shall be followed whenever applicable, noting that site-specific conditions or project-specific plans may require adjustments in methodology.

3.0 GLOSSARY

Direct Push Technology (DPT) - DPT refers to sampling tools and sensors that are driven directly into the ground without the use of conventional drilling equipment. DPT typically utilizes hydraulic pressure and/or percussion hammers to advance the sampling tools. A primary advantage of DPT over conventional drilling techniques is that DPT results in the generation of little or no investigation derived waste.

Geoprobe® - Geoprobe® is a manufacturer of a hydraulically-powered, percussion/probing machines utilizing DPT to collect subsurface environmental samples. Geoprobe® relies on a relatively small amount of static weight (vehicle) combined with percussion as the energy for advancement of a tool string. The Geoprobe® equipment can be mounted in a multitude of vehicles for access to all types of environmental sites.

HydroPunch™ - HydroPunch™ is a manufacturer of stainless steel and Teflon® sampling tools that are capable of collecting representative groundwater and/or soil samples without requiring the installation of a groundwater monitoring well or conventional soil boring. HydroPunch™ is an example of DPT sampling equipment.

Flame Ionization Detector (FID) - A portable instrument for the measurement of many combustible organic compounds and a few inorganic compounds in air at parts-per million levels. The basis for the detection is the ionization of gaseous species utilizing a flame as the energizing source.

Photo Ionization Detector (PID) - A portable instrument for the measurement of many combustible organic compounds and a few inorganic compounds in air at parts-per million levels. The basis for the detection is the ionization of gaseous species utilizing ultraviolet radiation as the energizing source.

4.0 RESPONSIBILITIES

Project Manager - The Project Manager is responsible for selecting and/or reviewing the appropriate DPT drilling procedure required to support the project objectives.

Field Operations Leader (FOL)- The FOL is primarily responsible for performing the DPT in accordance with the project-specific plan.

5.0 SOIL SAMPLING PROCEDURES

5.1 General

The common methodology for the investigation of the vadose zone is soil boring drilling and soil sampling. However, drilling soil borings can be very expensive. Generally the advantage of DPT for subsurface soil sampling is the reduced cost of disposal of drilling cuttings and shorter sampling times.

5.2 Sampling Equipment

Equipment needed for conducting DPT drilling for subsurface soil sampling includes, but is not limited to, the following:

- Geoprobe® Sampling Kit
- Cut-resistant gloves
- 4-foot x 1.5-inch diameter macrocore sampler
- Probe sampling adapters
- Roto-hammer with 1.5-inch bit
- Disposable acetate liners for soil macrocore sampler
- Cast aluminum or steel drive points
- Geoprobe® AT-660 Series Large Bore Soil Sampler, or equivalent
- Standard decontamination equipment and solutions

For health and safety equipment and procedures, follow the direction provided in the Safe Work Permit in Attachment 1, or the more detailed directions provided in the project's Health and Safety Plan.

5.3 DPT Sampling Methodology

There are several methods for the collection of soil samples using DPT drilling. The most common method is discussed in the following section. Variations of the following method may be conducted upon approval of the Project Manager in accordance with the project-specific plan.

- Macrocore samplers fitted with detachable aluminum or steel drive points are driven into the ground using hydraulic pressure. If there is concrete or pavement over a sampling location, a Roto-hammer is used to drill a minimum 1.5-inch diameter hole through the surface material. A Roto-hammer may also be used if very dense soils are encountered.

- The sampler is advanced continuously in 4-foot intervals or less if desired. No soil cuttings are generated because the soil which is not collected in the sampler is displaced within the formation.

- The sampler is retracted from the hole, and the 4-foot continuous sample is removed from the outer coring tube. The sample is contained within an inner acetate liner.

- Attach the metal trough from the Geoprobe® Sampling Kit firmly to the tail gate of a vehicle. If a vehicle with a tail gate is not available, secure the trough on another suitable surface.

- Place the acetate liner containing the soils in the trough.

- While wearing cut-resistant gloves (constructed of leather or other suitable material), cut the acetate liner through its entire length using the double-bladed knife that accompanies the Geoprobe® Sampling Kit. Then remove the strip of acetate from the trough to gain access to the collected soils. Do not attempt to cut the acetate liner while holding it in your hand.

- Field screen the sample with an FID or PID, and observe/examine the sample (according to SOP GH-1.3). If appropriate, transfer the sample to sample bottles for laboratory analysis. If additional volume is required, push an additional boring adjacent to the first and composite/mix the same interval. Field compositing is usually not acceptable for sample requiring volatile organics analysis.

- Once sampling has been completed, the hole is backfilled with bentonite chips or bentonite cement grout, depending upon project requirements. Asphalt or concrete patch is used to cap holes through paved or concrete areas. All holes should be finished smooth to existing grade.

- In the event the direct push van/truck cannot be driven to a remote location or a sampling location with difficult accessibility, sampling probes may be advanced and sampled manually or with air/electric operated equipment (e.g., jack hammer).

- Sampling equipment is decontaminated prior to collecting the next sample.

6.0 GROUNDWATER SAMPLING PROCEDURES

6.1 General

The most common methodology for the investigation of groundwater is the installation and sampling of permanent monitoring wells. If only groundwater screening is required, the installation and sampling of temporary well points may be performed. The advantage of temporary well point installation using DPT is reduced cost due to no or minimal disposal of drilling cuttings and well construction materials, and shorter installation/times sampling.

Two disadvantages of DPT drilling for well point installation are:

- In aquifers with low yields, well points may have to be sampled without purging or development.
- If volume requirements are high, this method can be time consuming for low yield aquifers.

6.2 Sampling Equipment

Equipment needed for temporary well installation and sampling using DPT includes, but is not limited, to the following:

- 2-foot x 1-inch diameter mill-slotted (0.005 to 0.02-inch) well point
- Connecting rods
- Roto-hammer with 1.5-inch bit
- Mechanical jack
- 1/4-inch OD polyethylene tubing
- 3/8-inch OD polyethylene tubing
- Peristaltic pump
- Standard decontamination equipment and solutions

6.3 DPT Temporary Well Point Installation and Sampling Methodology

There are several methods for the installation and sampling of temporary well points using DPT. The most common methodology is discussed below. Variations of the following method may be conducted upon approval of the Project Manager in accordance with the project specific plan.

- A 2-foot x 1-inch diameter mill-slotted (0.005 to 0.02-inch) well point attached to connecting rods is driven into the ground to the desired depth using a rotary electric hammer or other direct push drill rig. If there is concrete or pavement over a sampling location, a Roto-hammer or electric coring machine is used to drill a hole through the surface material.

- The well point will be allowed to equilibrate for at least 15 minutes, after which a measurement of the static water level will be taken. The initial measurement of the water level will be used to assess the amount of water which is present in the well point and to determine the amount of silt and sand infiltration that may have occurred.

- The well point will be developed using a peristaltic pump and polyethylene tubing to remove silt and sand which may have entered the well point. The well point is developed by inserting polyethylene tubing to the bottom of the well point and lifting and lowering the tubing slightly while the pump is operating. The pump will be operated at a maximum rate of approximately 2 liters per minute. After removal of sediment from the bottom of the well point, the well point will be vigorously pumped at maximum capacity until discharge water is visibly clear and no further sediments are being generated. Measurements of pH, specific conductance, temperature, and turbidity shall be recorded every 5 to 10 minutes during the purging process. After two consistent readings of pH, specific conductance, temperature and turbidity ($\pm$10 percent), the well may be sampled.

- A sample will be collected using the peristaltic pump set at the same or reduced speed as during well development. Samples (with the exception of the samples to be analyzed for volatile organic compounds, VOCs) will be collected directly from the pump discharge. Sample containers for VOCs will be filled by (first shutting off the pump) crimping the discharge end of the sample tubing when filled, removing the inlet end of the sample tubing from the well, suspending the inlet tubing above the vial, and allowing water to fill each vial by gravity flow.

- Once the groundwater sample has been collected, the connecting rods and well point will be removed from the hole with the direct push rig hydraulics. The hole will be backfilled with bentonite chips or bentonite cement grout, depending upon project requirements. Asphalt or concrete patch will be used to cap holes through paved or concrete areas. All holes will be finished smooth to existing grade.

- In the event the direct push van/truck cannot be driven to a remote location or sampling location with difficult accessibility, sampling probes may be advanced and sampled manually or with air/electric-operated equipment (e.g., jack hammer).

- Decontaminate the equipment before moving to the next location.

7.0 RECORDS

A record of all field procedures, tests, and observations must be recorded in the field logbook, boring logs, and sample log sheets, as needed. Entries should include all pertinent data regarding the investigation. The use of sketches and field landmarks will help to supplement the investigation and evaluation.

ATTACHMENT 1
SAFE WORK PERMIT FOR DPT OPERATIONS

Permit No. _____ Date: _____ Time: From _____ to _____

SECTION I: General Job Scope

I. Work limited to the following (description, area, equipment used): **Monitoring well drilling and installation through direct push technology**

II. Required Monitoring Instruments: _____

III. Field Crew: _____

IV. On-site Inspection conducted ☐ Yes ☐ No Initials of Inspector _____ _____ TtNUS

SECTION II: General Safety Requirements (To be filled in by permit issuer)

V. Protective equipment required

Level D ☒ Level B ☐
Level C ☐ Level A ☐
Detailed on Reverse

Respiratory equipment required

Full face APR	☐	Escape Pack	☐
Half face APR	☐	SCBA	☐
SKA-PAC SAR	☐	Bottle Trailer	☐
Skid Rig	☐	None	☒

Level D Minimum Requirements: Sleeved shirt and long pants, safety footwear, and work gloves. Safety glasses, hard hats, and hearing protection will be worn when working near or sampling in the vicinity of the DPT rig.

Modifications/Exceptions.

VI. Chemicals of Concern | Action Level(s) | Response Measures

VII. Additional Safety Equipment/Procedures

Hard-hat	☒ Yes ☐ No	Hearing Protection (Plugs/Muffs)	☒ Yes ☐ No
Safety Glasses	☒ Yes ☐ No	Safety belt/harness	☐ Yes ☒ No
Chemical/splash goggles	☐ Yes ☒ No	Radio	☐ Yes ☒ No
Splash Shield	☐ Yes ☒ No	Barricades	☒ Yes ☐ No
Splash suits/coveralls	☐ Yes ☐ No	Gloves (Type - _____)	☐ Yes ☐ No
Steel toe Work shoes or boots	☒ Yes ☐ No	Work/warming regimen	☐ Yes ☐ No

Modifications/Exceptions: Reflective vests for high traffic areas.

VIII. Procedure review with permit acceptors

	Yes	NA		Yes	NA
Safety shower/eyewash (Location & Use)	☐	☒	Emergency alarms	☐	☐
Daily tail gate meetings	☒	☐	Evacuation routes	☐	☐
Contractor tools/equipment/PPE inspected	☐	☐	Assembly points	☐	☐

IX. Site Preparation

Utility Clearances obtained for areas of subsurface investigation	☐ Yes ☐ No
Physical hazards removed or blockaded	☐ Yes ☐ No
Site control boundaries demarcated/signage	☐ Yes ☐ No

X. Equipment Preparation

	Yes	NA
Equipment drained/depressurized	☐	☒
Equipment purged/cleaned	☐	☒
Isolation checklist completed	☐	☒
Electrical lockout required/field switch tested	☐	☒
Blinds/misalignments/blocks & bleeds in place	☐	☒
Hazardous materials on walls/behind liners considered	☐	☒

XI. Additional Permits required (Hot work, confined space entry). ☐ Yes ☐ No
If yes, complete permit required or contact Health Sciences, Pittsburgh Office

XII. Special instructions, precautions:

Permit Issued by: _____ Permit Accepted by: _____

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		Effective Date 09/03	Revision 1
		Applicability Tetra Tech NUS, Inc.	
		Prepared Earth Sciences Department	
Subject AIR MONITORING AND SAMPLING		Approved D. Senovich	

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1.0 PURPOSE

The objective of this Standard Operating Procedure is to specify the proper approach and methodologies to identify and quantify airborne chemical contamination levels through the use of direct reading instrumentation and air sample collection. The results of these activities provide vital information for site characterization and risk assessment considerations.

2.0 SCOPE

Applies to all Tetra Tech NUS site activities where the potential for personnel exposures to respiratory health hazards exists.

3.0 GLOSSARY

Direct Reading Instruments (DRIs) - Instrumentation operating on various detection principles such as flame ionization or photoionization providing real time readings of ambient contaminants in air.

Personal/Area Air Sampling - Personal/area air sampling is conducted utilizing an air sampling pump and a specific collection media to quantify airborne contaminants.

Meteorological Considerations - Meteorological information must be collected on site to properly determine air sampling results, as well as aid in the characterization of contaminant potential plume migration and intensity. This information will also be used to support the selection of sampling locations and determine which samples should be analyzed. The meteorological information will be used to estimate downwind concentration levels based on short-term field levels encountered at the source.

4.0 RESPONSIBILITIES

Project Manager (PM) - Responsible for all aspects of project implementation and direction. The project manager is responsible for providing the necessary resources in support of all air monitoring and sampling applications.

Field Operations Leader (FOL) - Responsible for implementing the air monitoring program as detailed in approved project plans for the specific site. Air monitoring requirements will be included in both the Field Sampling and Analysis Plan (FSAP) and the site-specific Health and Safety Plan (HASP).

Health and Safety Officer (HSO) - The health and safety officer provides technical assistance to the FOL concerning air monitoring and sampling applications, collection methodologies, data interpretations, and establishes action items based on results. This information is further used to assess atmospheric migration of airborne chemical contaminants.

5.0 PROCEDURES

5.1 Introduction

Air monitoring is used to help establish criteria for worker safety, document potential exposures, and determine protective measures for the site personnel and the surrounding public. To accomplish this, it is necessary for an effective air surveillance program to be tailored to meet the conditions found at each work site.

During site operations, data are collected concerning air contaminants representative for site operations. Monitoring for vapors, gases, and particulates is performed using DRIs, air sampling systems, and

meteorological considerations. DRIs can be used to detect many organics as well as a few inorganics and can provide approximate total concentrations through applications of relative response ratios of contaminants to reference standards. If specific chemicals (organics and inorganics) have been identified, then properly calibrated DRIs can be used for more accurate onsite assessments.

The most accurate method for evaluating any air contaminant is to collect samples and analyze them at a qualified laboratory. Although accurate, this method presents two disadvantages: (1) cost and (2) the time required to obtain results. Analyzing large numbers of laboratory samples can be expensive, especially if results are needed quickly. Onsite laboratories tend to reduce the turnaround time, but unless they can analyze other types of samples, they may also be costly. In emergencies, time is often not available for laboratory analysis of samples either on site or off site.

To obtain air monitoring data rapidly at the site, DRI utilizing flame ionization detectors (FIDs), photoionization detectors (PIDs), and other detection methodology can be used. Some of these may be used as survey instruments or operated as gas chromatographs. As gas chromatographs, these instruments can provide real-time, qualitative/quantitative data when calibrated with standards of known air contaminants. Combined with selective laboratory analysis of samples, they provide a tool for evaluating airborne organic hazards on a real-time basis and at a lower cost than analyzing samples in a laboratory.

5.2 Air Sampling

For more complete information about air contaminants, measurements obtained with DRIs can be supplemented by collecting and analyzing air samples. To assess air contaminants more thoroughly, air sampling devices equipped with appropriate collection media may be placed at various locations throughout the area and on persons with at-risk occupations. These samples provide air quality information for the period of time they are taken, and can indicate contaminant types and concentrations over the sampling period. As a result, careful selection of sampling types, numbers, and locations, by a qualified health and safety professional is essential to obtain representative information. As data is obtained (from the analysis of samples, DRIs, knowledge about materials involved, site operations, and the potential for airborne toxic hazards), adjustments can be in the types of samples, number of samples collected, frequency of sampling, and analysis required. In addition to air samplers, area monitoring stations may also include DRIs equipped with data logging capabilities and operated as continuous air monitors.

Area air sampling locations may be located in various places as required by project and site needs. Area air sampling stations may include, but are not limited to:

- Upwind - Industrial operations, vehicle traffic, spills and other contributing sources may cause or otherwise result in the generation of air pollutants. Upwind samples establish background levels

- Support Zone (SZ) - Samples may be taken near the command post or other support facilities to ensure that they are, in fact, located in an unaffected area, and that the area remains clean throughout operations at the site.

- Contamination Reduction Zone (CRZ) - Air samples may be collected along the decontamination line to ensure that decontamination workers are properly protected and that onsite workers are not removing their respiratory protective gear in a contaminated area.

- Exclusion Zone (EZ) - The Exclusion Zone presents the greatest risk of release/generation of contaminants and requires the highest concern for air sampling. The location of sampling stations shall be based upon factors such as hot-spots detected by DRIs, types of substances present, and potential for airborne contaminants. The data from these stations, in conjunction with intermittent

walk-around surveys with DRIs, are used to verify the selection of proper levels of worker protection and EZ boundaries as well as to provide a continual record of air contaminants.

- Downwind - One or more sampling stations may be located downwind from the site to indicate if any air contaminants are leaving the site. If there are indications of airborne hazards in populated areas, appropriate response action must be taken and additional samplers should be placed downwind. Downwind locations are further determined based on meteorological considerations concerning generation, air plume migration, and intensity.

5.3 Media for Collecting Air Samples

Hazardous material incidents and abandoned waste sites can involve thousands of potentially dangerous substances, such as gases, vapors, and particulates that could become airborne. A variety of media are used to collect these substances. Sampling systems typically include a calibrated air sampling pump, which draws air into selected collection media. It is essential that appropriate, approved air sampling methodologies (such as those published by NIOSH, OSHA, and EPA) be followed for the collection of each specific analyte. Some of the most common types of samples and the collection media used for them are described in the following information:

One of the most common types of collection media is activated carbon which is an excellent adsorbent for most organic vapors. However, other solid adsorbents (such as Tenax, silica gel, and Florisil) are routinely used to sample specific organic compounds or classes of compounds that do not adsorb or desorb well onto activated carbon. To avoid stocking a large number of sorbents for collecting samples for various chemicals, a smaller number is generally chosen for collecting the widest range of materials or for chemicals known to be present. The vapors are collected using an industrial hygiene personal sampling pump with either one sampling port or a manifold capable of simultaneously collecting samples on several sorbent tubes (provided that sampling parameters such as flow rates and sample volumes are satisfied). For example, in a manifold with four sorbent tubes (or on individual pumps with varying flow rates), the tubes might contain:

- Activated carbon to collect vapors of materials with a boiling point above zero degrees Centigrade. Common materials collected on activated carbon include organic vapors such as solvents, BTEX, and ketones.

- A porous polymer, such as Tenax or Chromosorb, to collect substances (such as high-molecular-weight hydrocarbons, organophosphorus compounds, and the vapors of certain pesticides) that adsorb poorly onto activated carbon. Some of these porous polymers also absorb organic materials at low ambient temperatures more efficiently than carbon.

- A polar sorbent, such as silica gel, to collect organic vapors (aromatic amines, for example) that exhibit a relatively high dipole moment.

- Another specialty absorbent selected for the specific site. For example, a Florisil tube could be used if polychlorinated biphenyls are expected.

- Liquid impingers - aldehydes, ketones, phosgene, phenols.

- Glass fiber filters, membrane filters, Teflon filters - Inorganics and other semivolatile compounds.

- Airborne particulates can be either solid or liquid. Examples of common particulate analytes include some metals, fibers such as asbestos, and condensed particulates such as welding fumes. Dusts, fumes, smoke, and fibers are dispersed solids; mists and fogs are dispersed liquids. For air sampling, most particulates are collected using glass fiber, mixed cellulose ester, or polyvinyl chloride filters,

depending on the filter's ability to collect the subject material and its suitability for laboratory analysis. A cyclone is used to collect particles of respirable size. Atomic Absorption Spectrophotometry, Emission Spectroscopy, Phase Contrast Microscopy, and other techniques are used to analyze various types of particulates. Direct-reading monitors are also used to quantify particulate concentrations, and are usually based on the light-scattering properties of the particulate matter.

5.3.1 Other Methods

Colorimetric detector tubes can also be used with a sampling pump when monitoring for some specific compounds. A detector tube is a vial that contains a chemical preparation that reacts with the measured substance by changing color. Most detector tubes are scale tubes that permit a comparison of the length of the stain to an indicated concentration. Passive organic vapor monitors can be substituted for the active monitoring if they are available for the types of materials suspected to be present at a given site.

5.3.2 NIOSH Methods

The National Institute for Occupational Safety and Health's (NIOSH) Manual of Analytical Methods, 4th ed., contains acceptable methods for collecting and analyzing air samples for a variety of chemical substances. Consult these volumes for specific procedures.

5.4 Collection and Analysis

Collection and analysis of air samples is a multi-faceted task, and is part of the overall air surveillance program. The program is structured to cover the following air pathway analyses:

5.4.1 Selecting Monitoring Constituents

Applications within this program are accomplished using two considerations:

- Air surveillance for specific constituents is based on quantity of the pollutant and the likelihood for vapor release or generation.

- Controlling toxicity - These substances, even when represented in limited quantities, present the greatest threat to the public or worker safety, and influence environmental impact.

5.4.2 Specifying Meteorological Considerations

The following factors will influence sample collection:

- Wind direction and speed
- Sigma theta (atmospheric stability)
- Temperature
- Barometric pressure
- Humidity

These factors will provide information essential to properly arrive at accurate air sampling concentration results. This information is also used to identify how airborne chemical contaminants will react for modeling and for monitoring purposes. The results will provide indicators of plume movement, intensity, and dilution.

5.4.3 Design of Monitoring Network

The air surveillance network is structured to consider:

- Source characteristics (physical state; vapor release and/or generation; emission rates; and disturbance of the source impacting these aspects)
- Receptor sites (receptor sites are monitored and tracked based on priority)
- Meteorological consideration
- Air modeling input
- Data quality objectives

5.4.4 Air Monitoring Documentation/Data Reduction

5.4.4.1 Air Monitoring Documentation

Elements of the air surveillance program are used to provide documentation valuable to safely performing/containing site activities.

Air monitoring results from DRIs must be recorded, such as on instrument results reporting forms, or in the field logbook. This information, where applicable, will be correlated to air sampling information if/when collected.

Air sampling results for personnel and area measurement efforts must be validated, prior to notifying affected individuals. Personal air sampling results notification is accomplished through verbal or written communications.

Results of air monitoring/sampling activities can be identified on site maps. This information is used to structure operational zones and identify levels of protection.

5.4.4.2 Data Reduction

Data reduction combines and correlates the DRI results, air sampling results, and meteorological information to determine area and source airborne contaminant levels and movement.

All air sampling surveillance efforts must incorporate appropriate and approved NIOSH, OSHA, or EPA analytical methods. These procedures identify specific sample collection media, sampling methodologies, and analytical procedures. Sample analysis for health and safety considerations must be further supported by using American Industrial Hygiene Association accredited laboratories.

5.5 Personnel Monitoring

In addition to area atmospheric sampling, personnel monitoring -- both active and passive -- can be used to sample for air contaminants. Representative workers must be identified, and equipped with appropriate personal sampling systems to determine contaminants at specific locations or for specific work being performed. When sampling devices are placed on workers (generally within 1 foot of the mouth and nose) the results are used to indicate worker exposures.

5.6 Calibration

As a rule, the entire air sampling system shall be calibrated. Proper pre-and post-calibration activities are essential for correct operation and for accurate data. In some instances, additional calibration during the sampling period may be required. The overall frequency of calibration will depend upon the particular sampling event, including the general handling and use of a given sampling system. Pump mechanisms shall be calibrated after repair, when newly purchased, and following suspected abuse. All DRIs will be calibrated according to manufacturers instructions. All calibration activities for both air monitoring and sampling equipment must be properly documented, such as through the use of a calibration form. This form will be kept on site throughout the life of the project. The calibration log will be submitted as documentation that instrument calibration was performed on a regular basis.

5.7 Meteorological Considerations

Meteorological information is an integral part of an air surveillance program. Data concerning wind speed and direction, temperature, barometric pressure, and humidity (singularly or in combination) are needed for:

- Selecting air sampling locations
- Calculating accurate air sampling results
- Calculating air dispersion
- Calibrating instruments
- Determining population at risk or environmental exposure from airborne contaminants

Knowledge of wind speed and direction is necessary to effectively place air samplers. In source-oriented ambient air sampling, samplers need to be located downwind (at different distances) of the source and others need to be placed to collect background samples. Shifts in wind direction must be known. Consequently, the samplers must be relocated or corrections made for these shifts. In addition, atmospheric simulation models for predicting contaminant dispersion and concentration need windspeed and direction as inputs for predictive calculations. Information may be needed concerning the frequency and intensity that winds blow from certain directions (windrose data). Consequently, the wind direction must be continually monitored when use of this type of data is contemplated.

Air sampling systems need to be calibrated before use. This must include corrections in the calibration curves for actual temperatures and pressures during the sampling event. After sampling, collected air volumes are also mathematically corrected for temperature and pressure conditions.

Air sampling is sometimes designed to assess population exposure (and frequently potential worker exposure). Air samplers are generally located in population centers, irrespective of wind direction. Even in these instances, however, meteorological data is needed for air dispersion modeling. Models are then used to predict or verify population-oriented sampling results.

Proper data is collected by having meteorological stations on site or by obtaining the information from one or more of several government or private organizations, which routinely collect this data. The choice of how information is obtained depends on the availability of reliable data at the location desired, resources needed to obtain meteorological equipment, accuracy of information needed, and use of information.

The collection, handling, and analysis of air samples is an intricate, involved process. Appropriate methodologies, media, and equipment must be used to collect accurate data. Furthermore, selection of appropriate numbers, types, and locations of samples is essential if the data collected are to be used for personnel exposure criteria. For these reasons, air sampling activities must be coordinated and conducted by properly qualified and experienced industrial hygiene professionals. Air monitoring activities also need to be established and monitored carefully. However, as the proper use of these instruments is

not as complicated as air sampling, it is commonly acceptable to cross-train capable environmental professionals to use DRIs, with adequate technical support provided by health and safety professionals.

6.0 REFERENCES

Standard Operating Safety Guides, EPA, November 1984.
NIOSH Manual of Analytical Methods, 4th Edition.

7.0 ATTACHMENTS

None.

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		Effective Date 02/04	Revision 3
		Applicability Tetra Tech NUS, Inc.	
		Prepared Earth Sciences Department	
Subject NON-RADIOLOGICAL SAMPLE HANDLING		Approved D. Senovich	

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1.0 PURPOSE

The purpose of this Standard Operating Procedure (SOP) is to provide information on sample preservation, packaging, and shipping procedures to be used in handling environmental samples submitted for chemical constituent, biological, or geotechnical analysis. Sample chain-of-custody procedures and other aspects of field documentation are addressed in SOP SA-6.3. Sample identification is addressed in SOP CT-04.

2.0 SCOPE

This procedure describes the appropriate containers to be used for samples depending on the analyses to be performed, and the steps necessary to preserve the samples when shipped off site for chemical analysis.

3.0 GLOSSARY

Hazardous Material - A substance or material which has been determined by the Secretary of Transportation to be capable of posing an unreasonable risk to health, safety, and property when transported in commerce, and which has been so designated. Under 49 CFR, the term includes hazardous substances, hazardous wastes, marine pollutants, and elevated temperature materials, as well as materials designated as hazardous under the provisions of §172.101 and §172.102 and materials that meet the defining criteria for hazard classes and divisions in Part 173. With slight modifications, IATA has adopted DOT "hazardous materials" as IATA "Dangerous Goods."

Hazardous Waste - Any substance listed in 40 CFR, Subpart D (ỿ261.30 et seq.), or otherwise characterized as ignitable, corrosive, reactive, or toxic (as defined by Toxicity Characteristic Leaching Procedure, TCLP, analysis) as specified under 40 CFR, Subpart C (ỿ261.20 et seq.), that would be subject to manifest requirements specified in 40 CFR 262. Such substances are defined and regulated by EPA.

Marking - A descriptive name, identification number, instructions, cautions, weight, specification or UN marks, or combination thereof required on outer packaging of hazardous materials.

n.o.i - Not otherwise indicated (may be used interchangeably with n.o.s.).

n.o.s. - Not otherwise specified.

Packaging - A receptacle and any other components or materials necessary for compliance with the minimum packaging requirements of 49 CFR 174, including containers (other than freight containers or overpacks), portable tanks, cargo tanks, tank cars, and multi-unit tank-car tanks to perform a containment function in conformance with the minimum packaging requirements of 49 CFR 173.24(a) & (b).

Placard - Color-coded, pictorial sign which depicts the hazard class symbol and name and which is placed on the side of a vehicle transporting certain hazardous materials.

Common Preservatives:

- Hydrochloric Acid - HCl
- Sulfuric Acid - H_2SO_4
- Nitric Acid - HNO_3
- Sodium Hydroxide - NaOH

Other Preservatives

- Zinc Acetate
- Sodium Thiosulfate - $Na_2S_2O_3$

Normality (N) - Concentration of a solution expressed as equivalent per liter, an equivalent being the amount of a substance containing 1 gram-atom of replaceable hydrogen or its equivalent.

Reportable Quantity (RQ) - For the purposes of this SOP, means the quantity specified in column 3 of the Appendix to DOT 49 CFR §172.101 for any material identified in column 1 of the appendix. A spill greater than the amount specified must be reported to the National Response Center.

Sample - A sample is physical evidence collected from a facility or the environment, which is representative of conditions at the location and time of collection.

4.0 RESPONSIBILITIES

Field Operations Leader - Directly responsible for the bottling, preservation, labeling, packaging, shipping, and custody of samples up to and including release to the shipper.

Field Samplers - Responsible for initiating the Chain-of-Custody Record (per SOP SA-6.3), implementing the packaging and shipping requirements, and maintaining custody of samples until they are relinquished to another custodian or to the shipper.

5.0 PROCEDURES

Sample identification, labeling, documentation, and chain-of-custody are addressed by SOP SA-6.3.

5.1 Sample Containers

Different types of chemicals react differently with sample containers made of various materials. For example, trace metals adsorb more strongly to glass than to plastic, whereas many organic chemicals may dissolve various types of plastic containers. Attachments A and B show proper containers (as well as other information) per 40 CFR 136. In general, the sample container shall allow approximately 5-10 percent air space ("ullage") to allow for expansion/vaporization if the sample warms during transport. However, for collection of volatile organic compounds, head space shall be omitted. The analytical laboratory will generally provide certified-clean containers for samples to be analyzed for chemical constituents. Shelby tubes or other sample containers are generally provided by the driller for samples requiring geotechnical analysis. Sufficient lead time shall be allowed for a delivery of sample container orders. Therefore, it is critical to use the correct container to maintain the integrity of the sample prior to analysis.

Once opened, the container must be used at once for storage of a particular sample. Unused but opened containers are to be considered contaminated and must be discarded. Because of the potential for introduction of contamination, they cannot be reclosed and saved for later use. Likewise, any unused containers which appear contaminated upon receipt, or which are found to have loose caps or a missing Teflon liner (if required for the container), shall be discarded.

5.2 Sample Preservation

Many water and soil samples are unstable and therefore require preservation to prevent changes in either the concentration or the physical condition of the constituent(s) requiring analysis. Although complete and irreversible preservation of samples is not possible, preservation does retard the chemical and biological

changes that inevitably take place after the sample is collected. Preservation techniques are usually limited to pH control, chemical addition(s), and refrigeration/ freezing (certain biological samples only).

5.2.1 Overview

The preservation techniques to be used for various analytes are listed in Attachments A and B. Reagents required for sample preservation will either be added to the sample containers by the laboratory prior to their shipment to the field or be added in the field (in a clean environment). Only high purity reagents shall be used for preservation. In general, aqueous samples of low-concentration organics (or soil samples of low- or medium-concentration organics) are cooled to 4°C. Medium-concentration aqueous samples, high-hazard organic samples, and some gas samples are typically not preserved. Low-concentration aqueous samples for metals are acidified with HNO_3, whereas medium-concentration and high-hazard aqueous metal samples are not preserved. Low- or medium-concentration soil samples for metals are cooled to 4°C, whereas high-hazard samples are not cooled.

The following subsections describe the procedures for preparing and adding chemical preservatives. Attachments A and B indicate the specific analytes which require these preservatives.

The FOL is responsible for ensuring that an accurate Chemical Inventory is created and maintained for all hazardous chemicals brought to the work site (see Section 5 of the TtNUS Health and Safety Guidance Manual). Furthermore, the FOL must ensure that a corresponding Material Safety Data Sheet (MSDS) is collected for every substance entered on the site Chemical Inventory, and that all persons using/handling/ disposing of these substances review the appropriate MSDS for substances they will work with. The Chemical Inventory and the MSDSs must be maintained at each work site in a location and manner where they are readily-accessible to all personnel.

5.2.2 Preparation and Addition of Reagents

Addition of the following acids or bases may be specified for sample preservation; these reagents shall be analytical reagent (AR) grade or purer and shall be diluted to the required concentration with deionized water before field sampling commences. To avoid uncontrolled reactions, be sure to Add Acid to water (not vice versa). A dilutions guide is provided below.

Acid/Base	Dilution	Concentration	Estimated Amount Required for Preservation
Hydrochloric Acid (HCl)	1 part concentrated HCl: 1 part double-distilled, deionized water	6N	5-10 mL
Sulfuric Acid (H_2SO_4)	1 part concentrated H_2SO_4: 1 part double-distilled, deionized water	18N	2 - 5 mL
Nitric Acid (HNO_3)	Undiluted concentrated HNO_3	16N	2 - 5 mL
Sodium Hydroxide (NaOH)	400 grams solid NaOH dissolved in 870 mL double-distilled, deionized water; yields 1 liter of solution	10N	2 mL

The amounts required for preservation shown in the above table assumes proper preparation of the preservative and addition of the preservative to one liter of aqueous sample. This assumes that the sample is initially at pH 7, is poorly buffered, and does not contain particulate matter; as these conditions vary, more preservative may be required. Consequently, the final sample pH must be checked using narrow-range pH paper, as described in the generalized procedure detailed below:

- Pour off 5-10 mL of sample into a dedicated, clean container. Use some of this sample to check the initial sample pH using wide range (0-14) pH paper. Never dip the pH paper into the sample; always apply a drop of sample to the pH paper using a clean stirring rod or pipette.

- Add about one-half of the estimated preservative required to the original sample bottle. Cap and invert gently several times to mix. Check pH (as described above) using medium range pH paper (pH 0-6 or pH 7.5-14, as applicable).

- Cap sample bottle and seal securely.

Additional considerations are discussed below:

- To test if ascorbic acid must be used to remove oxidizing agents present in the sample before it can be properly preserved, place a drop of sample on KI-starch paper. A blue color indicates the need for ascorbic acid addition.

  If required, add a few crystals of ascorbic acid to the sample and retest with the KI-starch paper. Repeat until a drop of sample produces no color on the KI-starch paper. Then add an additional 0.6 grams of ascorbic acid per each liter of sample volume.

  Continue with proper base preservation of the sample as described above.

- Samples for sulfide analysis must be treated by the addition of 4 drops (0.2 mL) of 2N zinc acetate solution per 100 ml of sample.

  The 2N zinc acetate solution is made by dissolving 220 grams of zinc acetate in 870 mL of double-distilled, deionized water to make 1 liter of solution.

  The sample pH is then raised to 9 using the NaOH preservative.

- Sodium thiosulfate must be added to remove residual chlorine from a sample. To test the sample for residual chlorine use a field test kit specially made for this purpose.

  If residual chlorine is present, add 0.08 grams of sodium thiosulfate per liter of sample to remove the residual chlorine.

  Continue with proper acidification of the sample as described above.

For biological samples, 10% buffered formalin or isopropanol may also be required for preservation. Questions regarding preservation requirements should be resolved through communication with the laboratory before sampling begins.

5.3 Field Filtration

At times, field-filtration may be required to provide for the analysis of dissolved chemical constituents. Field-filtration must be performed prior to the preservation of samples as described above. General procedures for field filtration are described below:

- The sample shall be filtered through a non-metallic, 0.45-micron membrane filter, immediately after collection. The filtration system shall consist of dedicated filter canister, dedicated tubing, and a peristaltic pump with pressure or vacuum pumping squeeze action (since the sample is filtered by mechanical peristalsis, the sample travels only through the tubing).

- To perform filtration, thread the tubing through the peristaltic pump head. Attach the filter canister to the discharge end of the silicon tubing (note flow direction arrow); attach the aqueous sample container to the intake end of the silicon tubing. Turn the peristaltic pump on and perform filtration. Run approximately 100 ml of sample through the filter and discard prior to sample collection.

- Continue by preserving the filtrate (contained in the filter canister), as applicable and generally described above.

5.4 Sample Packaging and Shipping

Only employees who have successfully completed the TtNUS "Shipping Hazardous Materials" training course are authorized to package and ship hazardous substances. These trained individuals are responsible for performing shipping duties in accordance with this training.

Samples collected for shipment from a site shall be classified as either environmental or hazardous material samples. Samples from drums containing materials other than Investigative Derived Waste (IDW) and samples obtained from waste piles or bulk storage tanks are generally shipped as hazardous materials. A distinction must be made between the two types of samples in order to:

- Determine appropriate procedures for transportation of samples (if there is any doubt, a sample shall be considered hazardous and shipped accordingly.)

- Protect the health and safety of transport and laboratory personnel receiving the samples (special precautions are used by the shipper and at laboratories when hazardous materials are received.)

Detailed procedures for packaging environmental samples are outlined in the remainder of this section.

5.4.1 Environmental Samples

Environmental samples are packaged as follows:

- Place properly identified sample container, with lid securely fastened, in a plastic bag (e.g. Ziploc baggie), and seal the bag.

- Place sample in a cooler constructed of sturdy material which has been lined with a large, plastic bag (e.g. "garbage" bag). Drain plugs on coolers must be taped shut.

- Pack with enough cushioning materials such as bubble wrap (shoulders of bottles must be iced if required) to minimize the possibility of the container breaking.

- If cooling is required (see Attachments A and B), place ice around sample container shoulders, and on top of packing material (minimum of 8 pounds of ice for a medium-size cooler).

- Seal (i.e., tape or tie top in knot) large liner bag.

- The original (top, signed copy) of the COC form shall be placed inside a large Ziploc-type bag and taped inside the lid of the shipping cooler. If multiple coolers are sent but are included on one COC form, the COC form should be sent with the cooler containing the vials for VOC analysis. The COC form should then state how many coolers are included with that shipment.

- Close and seal outside of cooler as described in SOP SA-6.3. Signed custody seals must be used.

Coolers must be marked as containing "Environmental Samples." The appropriate side of the container must be marked "This End Up" and arrows placed appropriately. No DOT marking or labeling is required; there are no DOT restrictions on mode of transportation.

6.0 REFERENCES

American Public Health Association, 1981. Standard Methods for the Examination of Water and Wastewater, 15th Edition. APHA, Washington, D.C.

International Air Transport Association (latest issue). Dangerous Goods Regulations, Montreal, Quebec, Canada.

U.S. Department of Transportation (latest issue). Hazardous Materials Regulations, 49 CFR 171-177.

U.S. EPA, 1984. "Guidelines Establishing Test Procedures for the Analysis of Pollutants under Clean Water Act." Federal Register, Volume 49 (209), October 26, 1984, p. 43234.

U.S. EPA, 1979. Methods for Chemical Analysis of Water and Wastes. EPA-600/4-79-020, U.S. EPA-EMSL, Cincinnati, Ohio.

ATTACHMENT A

GENERAL SAMPLE CONTAINER AND PRESERVATION REQUIREMENTS

Sample Type and Concentration			Container[1]	Sample Size	Preservation[2]	Holding Time[2]
WATER						
Organics (GC&GC/MS)	VOC	Low	Borosilicate glass	2 x 40 mL	Cool to 4°C HCl to ≤ 2	14 days[9]
	Extractables SVOCs and pesticide/PCBs)	(Low	Amber glass	2x2 L or 4x1 L	Cool to 4°C	7 days to extraction; 40 days after extraction
	Extractables SVOCs and pesticide/PCBs)	(Medium	Amber glass	2x2 L or 4x1 L	None	7 days to extraction; 40 days after extraction
Inorganics	Metals	Low	High-density polyethylene	1 L	HNO_3 to pH ≤2	6 months (Hg-28 days)
		Medium	Wide-mouth glass	16 oz.	None	6 months
	Cyanide	Low	High-density polyethylene	1 L	NaOH to pH>12	14 days
	Cyanide	Medium	Wide-mouth glass	16 oz.	None	14 days
Organic/ Inorganic	High Hazard		Wide-mouth glass	8 oz.	None	14 days
SOIL						
Organics (GC&GC/MS)	VOC		EnCore Sampler	(3) 5 g Samplers	Cool to 4°C	48 hours to lab preservation
	Extractables SVOCs and pesticides/PCBs)	(Low	Wide-mouth glass	8 oz.	Cool to 4°C	14 days to extraction; 40 days after extraction
	Extractables SVOCs and pesticides/PCBs)	(Medium	Wide-mouth glass	8 oz.	Cool to 4°C	14 days to extraction; 40 days after extraction
Inorganics	Low/Medium		Wide-mouth glass	8 oz.	Cool to 4°C	6 months (Hg - 28 days) Cyanide (14 days)
Organic/Inorganic	High Hazard		Wide-mouth glass	8 oz.	None	NA
Dioxin/Furan	All		Wide-mouth glass	4 oz.	None	35 days until extraction; 40 days after extraction
TCLP	All		Wide-mouth glass	8 oz.	None	7 days until preparation; analysis as per fraction
AIR						
Volatile Organics	Low/Medium		Charcoal tube -- 7 cm long, 6 mm OD, 4 mm ID	100 L air	Cool to 4°C	5 days recommended

1 All glass containers should have Teflon cap liners or septa.
2 See Attachment E. Preservation and maximum holding time allowances per 40 CFR 136.

ATTACHMENT B

ADDITIONAL REQUIRED CONTAINERS, PRESERVATION TECHNIQUES, AND HOLDING TIMES

Parameter Number/Name	Container[1]	Preservation[2][3]	Maximum Holding Time[4]
INORGANIC TESTS:			
Acidity	P, G	Cool, 4°C	14 days
Alkalinity	P, G	Cool, 4°C	14 days
Ammonia - Nitrogen	P, G	Cool, 4°C; H_2SO_4 to pH 2	28 days
Biochemical Oxygen Demand (BOD)	P, G	Cool, 4°C	48 hours
Bromide	P, G	None required	28 days
Chemical Oxygen Demand (COD)	P, G	Cool, 4°C; H_2SO_4 to pH 2	28 days
Chloride	P, G	None required	28 days
Chlorine, Total Residual	P, G	None required	Analyze immediately
Color	P, G	Cool, 4°C	48 hours
Cyanide, Total and Amenable to Chlorination	P, G	Cool, 4°C; NaOH to pH 12; 0.6 g ascorbic acid[5]	14 days[6]
Fluoride	P	None required	28 days
Hardness	P, G	HNO_3 to pH 2; H_2SO_4 to pH 2	6 months
Total Kjeldahl and Organic Nitrogen	P, G	Cool, 4°C; H_2SO_4 to pH 2	28 days
Nitrate - Nitrogen	P, G	None required	48 hours
Nitrate-Nitrite - Nitrogen	P, G	Cool, 4°C; H_2SO_4 to pH 2	28 days
Nitrite - Nitrogen	P, G	Cool, 4°C	48 hours
Oil & Grease	G	Cool, 4°C; H_2SO_4 to pH 2	28 days
Total Organic Carbon (TOC)	P, G	Cool, 4°C; HCl or H_2SO_4 to pH 2	28 days
Orthophosphate	P, G	Filter immediately; Cool, 4°C	48 hours
Oxygen, Dissolved-Probe	G Bottle & top	None required	Analyze immediately
Oxygen, Dissolved-Winkler	G Bottle & top	Fix on site and store in dark	8 hours
Phenols	G	Cool, 4°C; H_2SO_4 to pH 2	28 days
Phosphorus, Total	P, G	Cool, 4°C; H_2SO_4 to pH 2	28 days
Residue, Total	P, G	Cool, 4°C	7 days
Residue, Filterable (TDS)	P, G	Cool, 4°C	7 days
Residue, Nonfilterable (TSS)	P, G	Cool, 4°C	7 days
Residue, Settleable	P, G	Cool, 4°C	48 hours
Residue, Volatile (Ash Content)	P, G	Cool, 4°C	7 days
Silica	P	Cool, 4°C	28 days
Specific Conductance	P, G	Cool, 4°C	28 days
Sulfate	P, G	Cool, 4°C	28 days

ATTACHMENT B
ADDITIONAL REQUIRED CONTAINERS, PRESERVATION TECHNIQUES, AND HOLDING TIMES
PAGE TWO

Parameter Number/Name	Container[1]	Preservation[2][3]	Maximum Holding Time[4]
INORGANIC TESTS (Cont'd):			
Sulfide	P, G	Cool, 4°C; add zinc acetate plus sodium hydroxide to pH 9	7 days
Sulfite	P, G	None required	Analyze immediately
Turbidity	P, G	Cool, 4°C	48 hours
METALS:[7]			
Chromium VI (Hexachrome)	P, G	Cool, 4°C	24 hours
Mercury (Hg)	P, G	HNO_3 to pH 2	28 days
Metals, except Chromium VI and Mercury	P, G	HNO_3 to pH 2	6 months
ORGANIC TESTS:[8]			
Purgeable Halocarbons	G, Teflon-lined septum	Cool, 4°C; 0.008% $Na_2S_2O_3$[5]	14 days
Purgeable Aromatic Hydrocarbons	G, Teflon-lined septum	Cool, 4°C; 0.008% $Na_2S_2O_3$[5] HCl to pH 2 [9]	14 days
Acrolein and Acrylonitrile	G, Teflon-lined septum	Cool, 4°C; 0.008% $Na_2S_2O_3$[5] adjust pH to 4-5 [10]	14 days
Phenols[11]	G, Teflon-lined cap	Cool, 4°C; 0.008% $Na_2S_2O_3$[5]	7 days until extraction; 40 days after extraction
Benzidines[11],[12]	G, Teflon-lined cap	Cool, 4°C; 0.008% $Na_2S_2O_3$[5]	7 days until extraction[13]
Phthalate esters[11]	G, Teflon-lined cap	Cool, 4°C	7 days until extraction; 40 days after extraction
Nitrosamines[11],[14]	G, Teflon-lined cap	Cool, 4°C; store in dark; 0.008% $Na_2S_2O_3$[5]	7 days until extraction; 40 days after extraction
PCBs[11]	G, Teflon-lined cap	Cool, 4°C	7 days until extraction; 40 days after extraction
Nitroaromatics & Isophorone[11]	G, Teflon-lined cap	Cool, 4°C; 0.008% $Na_2S_2O_3$[5]; store in dark	7 days until extraction; 40 days after extraction
Polynuclear Aromatic Hydrocarbons (PAHs)[11],[14]	G, Teflon-lined cap	Cool, 4°C; 0.008% $Na_2S_2O_3$[5]; store in dark	7 days until extraction; 40 days after extraction
Haloethers[11]	G, Teflon-lined cap	Cool, 4°C; 0.008% $Na_2S_2O_3$[5]	7 days until extraction; 40 days after extraction
Dioxin/Furan (TCDD/TCDF)[11]	G, Teflon-lined cap	Cool, 4°C; 0.008% $Na_2S_2O_3$[5]	7 days until extraction; 40 days after extraction

ATTACHMENT B
ADDITIONAL REQUIRED CONTAINERS, PRESERVATION TECHNIQUES, AND HOLDING TIMES
PAGE THREE

(1) Polyethylene (P): generally 500 ml or Glass (G): generally 1L.
(2) Sample preservation should be performed immediately upon sample collection. For composite chemical samples each aliquot should be preserved at the time of collection. When use of an automated sampler makes it impossible to preserve each aliquot, then chemical samples may be preserved by maintaining at 4°C until compositing and sample splitting is completed.
(3) When any sample is to be shipped by common carrier or sent through the United States Mail, it must comply with the Department of Transportation Hazardous Materials Regulations (49 CFR Part 172).
(4) Samples should be analyzed as soon as possible after collection. The times listed are the maximum times that samples may be held before analysis and still be considered valid. Samples may be held for longer periods only if the permittee, or monitoring laboratory, has data on file to show that the specific types of samples under study are stable for the longer periods, and has received a variance from the Regional Administrator.
(5) Should only be used in the presence of residual chlorine.
(6) Maximum holding time is 24 hours when sulfide is present. Optionally, all samples may be tested with lead acetate paper before pH adjustments are made to determine if sulfide is present. If sulfide is present, it can be removed by the addition of cadmium nitrate powder until a negative spot test is obtained. The sample is filtered and then NaOH is added to pH 12.
(7) Samples should be filtered immediately on site before adding preservative for dissolved metals.
(8) Guidance applies to samples to be analyzed by GC, LC, or GC/MS for specific compounds.
(9) Sample receiving no pH adjustment must be analyzed within 7 days of sampling.
(10) The pH adjustment is not required if acrolein will not be measured. Samples for acrolein receiving no pH adjustment must be analyzed within 3 days of sampling.
(11) When the extractable analytes of concern fall within a single chemical category, the specified preservative and maximum holding times should be observed for optimum safeguard of sample integrity. When the analytes of concern fall within two or more chemical categories, the sample may be preserved by cooling to 4°C, reducing residual chlorine with 0.008% sodium thiosulfate, storing in the dark, and adjusting the pH to 6-9; samples preserved in this manner may be held for 7 days before extraction and for 40 days after extraction. Exceptions to this optional preservation and holding time procedure are noted in footnote 5 (re: the requirement for thiosulfate reduction of residual chlorine) and footnotes 12, 13 (re: the analysis of benzidine).
(12) If 1,2-diphenylthydrazine is likely to be present, adjust the pH of the sample to 4.0±0.2 to prevent rearrangement to benzidine.
(13) Extracts may be stored up to 7 days before analysis if storage is conducted under an inert (oxidant-free) atmosphere.
(14) For the analysis of diphenylnitrosamine, add 0.008% $Na_2S_2O_3$ and adjust pH to 7-10 with NaOH within 24 hours of sampling.
(15) The pH adjustment may be performed upon receipt at the laboratory and may be omitted if the samples are extracted within 72 hours of collection. For the analysis of aldrin, add 0.008% $Na_2S_2O_3$.

TABLE OF CONTENTS

1.0 PURPOSE

The purpose of this Standard Operating Procedure (SOP) is to identify and designate the field data record forms, logs and reports generally initiated and maintained for documenting Tetra Tech NUS field activities.

2.0 SCOPE

Documents presented within this procedure (or equivalents) shall be used for all Tetra Tech NUS field activities, as applicable. Other or additional documents may be required by specific client contracts or project planning documents.

3.0 GLOSSARY

None

4.0 RESPONSIBILITIES

Project Manager (PM) - The Project Manager is responsible for obtaining hardbound, controlled-distribution logbooks (from the appropriate source), as needed. In addition, the Project Manager is responsible for placing all field documentation used in site activities (i.e., records, field reports, sample data sheets, field notebooks, and the site logbook) in the project's central file upon the completion of field work.

Field Operations Leader (FOL) - The Field Operations Leader is responsible for ensuring that the site logbook, notebooks, and all appropriate and current forms and field reports illustrated in this guideline (and any additional forms required by the contract) are correctly used, accurately filled out, and completed in the required time-frame.

5.0 PROCEDURES

5.1 Site Logbook

5.1.1 General

The site logbook is a hard-bound, paginated, controlled-distribution record book in which all major onsite activities are documented. At a minimum, the following activities/events shall be recorded or referenced (daily) in the site logbook:

- All field personnel present
- Arrival/departure of site visitors
- Time and date of H&S training
- Arrival/departure of equipment
- Time and date of equipment calibration
- Start and/or completion of borehole, trench, monitoring well installation, etc.
- Daily onsite activities performed each day
- Sample pickup information
- Health and Safety issues (level of protection observed, etc.)
- Weather conditions

A site logbook shall be maintained for each project. The site logbook shall be initiated at the start of the first onsite activity (e.g., site visit or initial reconnaissance survey). Entries are to be made for every day

that onsite activities take place which involve Tetra Tech NUS or subcontractor personnel. Upon completion of the fieldwork, the site logbook must become part of the project's central file.

The following information must be recorded on the cover of each site logbook:

- Project name
- Tetra Tech NUS project number
- Sequential book number
- Start date
- End date

Information recorded daily in the site logbook need not be duplicated in other field notebooks (see Section 5.2), but must summarize the contents of these other notebooks and refer to specific page locations in these notebooks for detailed information (where applicable). An example of a typical site logbook entry is shown in Attachment A.

If measurements are made at any location, the measurements and equipment used must either be recorded in the site logbook or reference must be made to the field notebook in which the measurements are recorded (see Attachment A).

All logbook, notebook, and log sheet entries shall be made in indelible ink (black pen is preferred). No erasures are permitted. If an incorrect entry is made, the entry shall be crossed out with a single strike mark, and initialed and dated. At the completion of entries by any individual, the logbook pages used must be signed and dated. The site logbook must also be signed by the Field Operations Leader at the end of each day.

5.1.2 Photographs

When movies, slides, or photographs are taken of a site or any monitoring location, they must be numbered sequentially to correspond to logbook/notebook entries. The name of the photographer, date, time, site location, site description, and weather conditions must be entered in the logbook/notebook as the photographs are taken. A series entry may be used for rapid-sequence photographs. The photographer is not required to record the aperture settings and shutter speeds for photographs taken within the normal automatic exposure range. However, special lenses, films, filters, and other image-enhancement techniques must be noted in the logbook/notebook. If possible, such techniques shall be avoided, since they can adversely affect the accuracy of photographs. Chain-of-custody procedures depend upon the subject matter, type of camera (digital or film), and the processing it requires. Film used for aerial photography, confidential information, or criminal investigation require chain-of-custody procedures. Once processed, the slides of photographic prints shall be consecutively numbered and labeled according to the logbook/notebook descriptions. The site photographs and associated negatives and/or digitally saved images to compact disks must be docketed into the project's central file.

5.2 Field Notebooks

Key field team personnel may maintain a separate dedicated field notebook to document the pertinent field activities conducted directly under their supervision. For example, on large projects with multiple investigative sites and varying operating conditions, the Health and Safety Officer may elect to maintain a separate field notebook. Where several drill rigs are in operation simultaneously, each site geologist assigned to oversee a rig must maintain a field notebook.

5.3 Field Forms

All Tetra Tech NUS field forms (see list in Section 6.0 of this SOP) can be found on the company's intranet site (http://intranet.ttnus.com) under Field Log Sheets. Forms may be altered or revised for project-specific needs contingent upon client approval. Care must be taken to ensure that all essential information can be documented. Guidelines for completing these forms can be found in the related sampling SOP.

5.3.1 Sample Collection, Labeling, Shipment, Request for Analysis, and Field Test Results

5.3.1.1 Sample Log Sheet

Sample Log Sheets are used to record specified types of data while sampling. The data recorded on these sheets are useful in describing the sample as well as pointing out any problems, difficulties, or irregularities encountered during sampling. A log sheet must be completed for each sample obtained, including field quality control (QC) samples.

5.3.1.2 Sample Label

A typical sample label is illustrated in Attachment B. Adhesive labels must be completed and applied to every sample container. Sample labels can usually be obtained from the appropriate Program source electronically generated in-house, or are supplied from the laboratory subcontractor.

5.3.1.3 Chain-of-Custody Record Form

The Chain-of-Custody (COC) Record is a multi-part form that is initiated as samples are acquired and accompanies a sample (or group of samples) as they are transferred from person to person. This form must be used for any samples collected for chemical or geotechnical analysis whether the analyses are performed on site or off site. One carbonless copy of the completed COC form is retained by the field crew, one copy is sent to the Project Manager (or designee), while the original is sent to the laboratory. The original (top, signed copy) of the COC form shall be placed inside a large Ziploc-type bag and taped inside the lid of the shipping cooler. If multiple coolers are sent but are included on one COC form, the COC form should be sent with the cooler containing vials for VOC analysis or the cooler with the air bill attached. The air bill should then state how many coolers are included with that shipment. An example of a Chain-of-Custody Record form is provided as Attachment C. Once the samples are received at the laboratory, the sample cooler and contents are checked and any problems are noted on the enclosed COC form (any discrepancies between the sample labels and COC form and any other problems that are noted are resolved through communication between the laboratory point-of-contact and the Tetra Tech NUS Project Manager). The COC form is signed and copied. The laboratory will retain the copy while the original becomes part of the samples' corresponding analytical data package.

5.3.1.4 Chain-of-Custody Seal

Attachment D is an example of a custody seal. The Custody seal is an adhesive-backed label. It is part of a chain-of-custody process and is used to prevent tampering with samples after they have been collected in the field and sealed in coolers for transport to the laboratory. The COC seals are signed and dated by the sampler(s) and affixed across the lid and body of each cooler (front and back) containing environmental samples (see SOP SA-6.1). COC seals may be available from the laboratory; these seals may also be purchased from a supplier.

5.3.1.5 Geochemical Parameters Log Sheets

Field Analytical Log Sheets are used to record geochemical and/or natural attenuation field test results.

5.3.2 Hydrogeological and Geotechnical Forms

5.3.2.1 Groundwater Level Measurement Sheet

A Groundwater Level Measurement Sheet must be filled out for each round of water level measurements made at a site.

5.3.2.2 Data Sheet for Pumping Test

During the performance of a pumping test (or an in-situ hydraulic conductivity test), a large amount of data must be recorded, often within a short time period. The Pumping Test Data Sheet facilitates this task by standardizing the data collection format for the pumping well and observation wells, and allowing the time interval for collection to be laid out in advance.

5.3.2.3 Packer Test Report Form

A Packer Test Report Form must be completed for each well upon which a packer test is conducted.

5.3.2.4 Boring Log

During the progress of each boring, a log of the materials encountered, operation and driving of casing, and location of samples must be kept. The Summary Log of Boring, or Boring Log is used for this purpose and must be completed for each soil boring performed. In addition, if volatile organics are monitored on cores, samples, cuttings from the borehole, or breathing zone, (using a PID or FID), these readings must be entered on the boring log at the appropriate depth. The "Remarks" column can be used to subsequently enter the laboratory sample number, the concentration of key analytical results, or other pertinent information. This feature allows direct comparison of contaminant concentrations with soil characteristics.

5.3.2.5 Monitoring Well Construction Details Form

A Monitoring Well Construction Details Form must be completed for every monitoring well, piezometer, or temporary well point installed. This form contains specific information on length and type of well riser pipe and screen, backfill, filter pack, annular seal and grout characteristics, and surface seal characteristics. This information is important in evaluating the performance of the monitoring well, particularly in areas where water levels show temporal variation, or where there are multiple (immiscible) phases of contaminants. Depending on the type of monitoring well (in overburden or bedrock, stick-up or flush mount), different forms are used.

5.3.2.6 Test Pit Log

When a test pit or trench is constructed for investigative or sampling purposes, a Test Pit Log must be filled out by the responsible field geologist or sampling technician.

5.3.2.7 Miscellaneous Monitoring Well Forms

Monitoring Well Materials Certificate of Conformance should be used as the project directs to document all materials utilized during each monitoring well installation.

The Monitoring Well Development Record should be used as the project directs to document all well development activities.

5.3.2.8 Miscellaneous Field Forms - QA and Checklists

Container Sample and Inspection Sheet should be used as the project directs each time a container (drum, tank, etc.) is sampled and/or inspected.

QA Sample Log Sheet should be used at the project directs each time a QA sample is colleted, such as Rinsate Blank, Source Blank, etc.

Field Task Modification Request (FTMR) will be prepared for all deviations from the project planning documents. The FOL is responsible for initiating the FTMRs. Copies of all FTMRs will be maintained with the onsite planning documents and originals will be placed in the final evidence file.

The Field Project Daily Activities Check List and Field Project Pre-Mobilization Checklist should be used during both the planning and field effort to assure that all necessary tasks are planned for and completed. These two forms are not a requirement but a useful tool for most field work.

5.3.3 Equipment Calibration and Maintenance Form

The calibration or standardization of monitoring, measuring or test equipment is necessary to assure the proper operation and response of the equipment, to document the accuracy, precision or sensitivity of the measurement, and determine if correction should be applied to the readings. Some items of equipment require frequent calibration, others infrequent. Some are calibrated by the manufacturer, others by the user.

Each instrument requiring calibration has its own Equipment Calibration Log which documents that the manufacturer's instructions were followed for calibration of the equipment, including frequency and type of standard or calibration device. An Equipment Calibration Log must be maintained for each electronic measuring device used in the field; entries must be made for each day the equipment is used or in accordance with the manufacturer's recommendations.

5.4 Field Reports

The primary means of recording onsite activities is the site logbook. Other field notebooks may also be maintained. These logbooks and notebooks (and supporting forms) contain detailed information required for data interpretation or documentation, but are not easily useful for tracking and reporting of progress. Furthermore, the field logbook/notebooks remain onsite for extended periods of time and are thus not accessible for timely review by project management.

5.4.1 Daily Activities Report

To provide timely oversight of onsite contractors, Daily Activities Reports are completed and submitted as described below.

5.4.1.1 Description

The Daily Activities Report (DAR) documents the activities and progress for each day's field work. This report must be filled out on a daily basis whenever there are drilling, test pitting, well construction, or other related activities occurring which involve subcontractor personnel. These sheets summarize the work performed and form the basis of payment to subcontractors. The DAR form can be found on the TtNUS intranet site.

5.4.1.2 Responsibilities

It is the responsibility of the rig geologist to complete the DAR and obtain the driller's signature acknowledging that the times and quantities of material entered are correct.

5.4.1.3 Submittal and Approval

At the end of the shift, the rig geologist must submit the Daily Activities Report to the Field Operations Leader (FOL) for review and filing. The Daily Activities Report is not a formal report and thus requires no further approval. The DAR reports are retained by the FOL for use in preparing the site logbook and in preparing weekly status reports for submission to the Project Manager.

5.4.2 Weekly Status Reports

To facilitate timely review by project management, photocopies of logbook/notebook entries may be made for internal use.

It should be noted that in addition to summaries described herein, other summary reports may also be contractually required.

All Tetra Tech NUS field forms can be found on the company's intranet site at http://intranet.ttnus.com under Field Log Sheets.

6.0 LISTING OF TETRA TECH NUS FIELD FORMS FOUND ON THE TTNUS INTRANET SITE. HTTP://INTRANET.TTNUS.COM CLICK ON FIELD LOG SHEETS

Groundwater Sample Log Sheet
Surface Water Sample Log Sheet
Soil/Sediment Sample Log Sheet
Container Sample and Inspection Sheet
Geochemical Parameters (Natural Attenuation)
Groundwater Level Measurement Sheet
Pumping Test Data Sheet
Packer Test Report Form
Boring Log
Monitoring Well Construction Bedrock Flush Mount
Monitoring Well Construction Bedrock Open Hole
Monitoring Well Construction Bedrock Stick Up
Monitoring Well Construction Confining Layer
Monitoring Well Construction Overburden Flush Mount
Monitoring Well Construction Overburden Stick Up
Test Pit Log
Monitoring Well Materials Certificate of Conformance
Monitoring Well Development Record

Daily Activities Record
Field Task Modification Request
Hydraulic Conductivity Test Data Sheet
Low Flow Purge Data Sheet
QA Sample Log Sheet
Equipment Calibration Log
Field Project Daily Activities Checklist
Field Project Pre-Mobilization Checklist

ATTACHMENT A
TYPICAL SITE LOGBOOK ENTRY

START TIME: _____ DATE: _____

SITE LEADER: _____
PERSONNEL:

TtNUS	DRILLER	SITE VISITORS
_____	_____	_____
_____	_____	_____
_____	_____	_____

WEATHER: Clear, 68°F, 2-5 mph wind from SE

ACTIVITIES:

1. Steam jenney and fire hoses were set up.
2. Drilling activities at well ____ resumes. Rig geologist was _____. See Geologist's Notebook, No. 1, page 29-30, for details of drilling activity. Sample No. 123-21-S4 collected; see sample logbook, page 42. Drilling activities completed at 11:50 and a 4-inch stainless steel well installed. See Geologist's Notebook, No. 1, page 31, and well construction details for well _____.
3. Drilling rig No. 2 steam-cleaned at decontamination pit. Then set up at location of well _____.
4. Well _____ drilled. Rig geologist was _____. See Geologist's Notebook, No. 2, page ____ for details of drilling activities. Sample numbers 123-22-S1, 123-22-S2, and 123-22-S3 collected; see sample logbook, pages 43, 44, and 45.
5. Well _____ was developed. Seven 55-gallon drums were filled in the flushing stage. The well was then pumped using the pitcher pump for 1 hour. At the end of the hour, water pumped from well was "sand free."
6. EPA remedial project manger arrives on site at 14:25 hours.
7. Large dump truck arrives at 14:45 and is steam-cleaned. Backhoe and dump truck set up over test pit _____.
8. Test pit _____ dug with cuttings placed in dump truck. Rig geologist was _____. See Geologist's Notebook, No. 1, page 32, for details of test pit activities. Test pit subsequently filled. No samples taken for chemical analysis. Due to shallow groundwater table, filling in of test pit ___ resulted in a very soft and wet area. A mound was developed and the area roped off.
9. Express carrier picked up samples (see Sample Logbook, pages 42 through 45) at 17:50 hours. Site activities terminated at 18:22 hours. All personnel off site, gate locked.

_____

Field Operations Leader

ATTACHMENT B

Tetra Tech NUS, Inc. 661 Andersen Drive Pittsburgh, 15220 (412)921-7090	**Project:** **Site:** **Location:**	
Sample No:		**Matrix:**
Date:	**Time:**	**Preserve:**
Analysis:		
Sampled by:	**Laboratory:**	

ATTACHMENT C

TETRA TECH NUS, INC. CHAIN OF CUSTODY | NUMBER 3413 | PAGE ___ OF ___

PROJECT NO:	FACILITY:	PROJECT MANAGER	PHONE NUMBER	LABORATORY NAME AND CONTACT:
SAMPLERS (SIGNATURE)		FIELD OPERATIONS LEADER	PHONE NUMBER	ADDRESS
		CARRIER/WAYBILL NUMBER		CITY, STATE

STANDARD TAT ☐
RUSH TAT ☐
☐ 24 hr. ☐ 48 hr. ☐ 72 hr. ☐ 7 day ☐ 14 day

DATE / YEAR	TIME	SAMPLE ID	LOCATION ID	TOP DEPTH (FT)	BOTTOM DEPTH (FT)	MATRIX (GW, SO, SW, SD, QC, ETC.)	COLLECTION METHOD GRAP (G) COMP (C)	No. OF CONTAINERS	CONTAINER TYPE PLASTIC (P) or GLASS (G) / PRESERVATIVE USED / TYPE OF ANALYSIS								COMMENTS

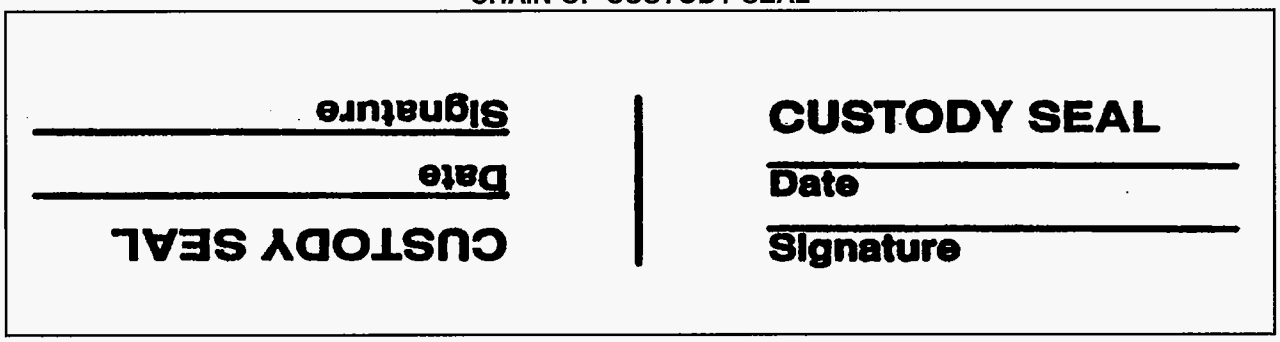
1. RELINQUISHED BY	DATE	TIME	1. RECEIVED BY	DATE	TIME
2. RELINQUISHED BY	DATE	TIME	2. RECEIVED BY	DATE	TIME
3. RELINQUISHED BY	DATE	TIME	3. RECEIVED BY	DATE	TIME
COMMENTS					

DISTRIBUTION: WHITE (ACCOMPANIES SAMPLE) YELLOW (FIELD COPY) PINK (FILE COPY) 4/02R

FORM NO. TtNUS-001

ATTACHMENT D

CHAIN-OF-CUSTODY SEAL



TETRA TECH	STANDARD OPERATING PROCEDURES	Number SA-7.1	Page 1 of 16
		Effective Date 01/28/2009	Revision 6
		Applicability Tetra Tech NUS, Inc.	
		Prepared Earth Sciences Department	
Subject DECONTAMINATION OF FIELD EQUIPMENT		Approved Tom Johnston	

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SECTION **PAGE**

ATTACHMENTS

1.0 PURPOSE

Decontamination is the process of removing and/or neutralizing site contaminants that have contacted and/or accumulated on equipment. The purpose of this Standard Operating Procedure (SOP) is to protect site personnel, the general public, and the environment while preserving or maintaining sample integrity. It is further intended through this procedure to describe the steps necessary for proper decontamination of drilling equipment, earth-moving equipment, chemical sampling equipment and field operation and analytical equipment.

2.0 SCOPE AND APPLICABILITY

This procedure applies to all equipment used to provide access to/acquire environmental samples that may have become contaminated through direct contact with contaminated media including air, water, and soil. This equipment includes drilling and heavy equipment and chemical sampling and field analytical equipment. Where technologically and economically feasible, single-use sealed disposable equipment will be employed to minimize the potential for cross-contamination. This SOP also provides general reference information on the control of contaminated materials.

Decontamination methods and equipment requirements may differ from one project to another. General equipment items are specified in Section 6.0, but project-specific equipment must be obtained to address the project-specific decontamination procedures presented in Section 7.0 and applicable subsections.

3.0 GLOSSARY

Alconox/Liquinox - A brand of phosphate-free laboratory-grade detergent.

Decontamination Solution - A solution selected/identified in the Health and Safety Plan or Project-Specific Quality Assurance Plan. The solution is selected and employed as directed by the project chemist/health and safety professional.

Deionized Water (DI) - Tap water that has been treated by passing through a standard deionizing resin column. This water may also pass through additional filtering media to attain various levels of analyte-free status. The DI water should meet College of American Pathologists (CAP) and National Committee for Clinical Laboratory Standards (NCCLS) specifications for reagent-grade Type I water.

Potable Water - Tap water from any municipal water treatment system. Use of an untreated potable water supply is not an acceptable substitute for tap water.

Pressure Washing - Process employing a high-pressure pump and nozzle configuration to create a high-pressure spray of potable water. High-pressure spray is employed to remove solids from equipment.

Solvent – A liquid in which solid chemicals or other liquids are dissolved. The solvent of choice is pesticide-grade isopropanol. Use of other solvents (methanol, acetone, or hexane) may be required for particular projects or for a particular purpose (e.g., removal of concentrated waste) and must be justified in the project planning documents. For example, it may be necessary to use hexane when analyzing for trace levels of pesticides, PCBs, or fuels. In addition, because many of these solvents are not miscible in water, the equipment should be air dried prior to use. Solvents should not be used on PVC equipment or well construction materials.

Steam Pressure Washing - A cleaning method employing a high-pressure spray of heated potable water to remove various organic/inorganic chemicals from equipment.

4.0 RESPONSIBILITIES AND PERSONNEL QUALIFICATIONS

Project Manager - Responsible for ensuring that all field activities are conducted in accordance with approved project plan(s) requirements.

Decontamination Personnel - Individuals assigned the task of decontamination. It is the responsibility of these individuals to understand the use and application of the decontamination process and solutions as well as the monitoring of that process to ensure that it is working properly. This is accomplished through visual evaluation, monitoring instrument scanning of decontaminated items, and/or through the collection of rinsate blanks to verify contaminant removal.

Field Operations Leader (FOL) - Responsible for the implementation of project-specific planning documents. This includes on-site verification that all field activities are performed in compliance with approved SOPs or as otherwise dictated by the approved project plan(s). The FOL is also responsible for the completion and accuracy of all field documentation.

Site Safety Officer (SSO) - Exercises shared responsibility with the FOL concerning decontamination effectiveness. All equipment arriving on site (as part of the equipment inspection), leaving the site, and moving between locations is required to go through a decontamination evaluation. This is accomplished through visual examination and/or instrument screening to determine the effectiveness of the decontamination process. Improper or incomplete decontamination is sufficient to restrict equipment from entering the site, exiting the site, or moving to a new location on the site until the objectives are successfully completed.

General personnel qualifications for decontamination activities include the following:

- Occupational Safety and Health Administration (OSHA) 40-hour and applicable refresher training.
- Capability of performing field work under the expected physical and environmental (i.e., weather) conditions.
- Familiarity with appropriate decontamination procedures.

5.0 HEALTH AND SAFETY

In addition to the health and safety issues and reminders specified in subsections of this SOP, the following considerations and requirements must be observed as SOPs for field equipment decontamination activities:

- If any solvents or hazardous chemicals (e.g., isopropyl alcohol) are to be used in equipment decontamination activities, the FOL must first obtain the manufacturer's/supplier's Material Safety Data Sheet (MSDS) and assure that it is reviewed by all users (prior to its use), added to the site Hazardous Chemical Inventory, and maintained on site as part of the project Hazard Communication Program.
- Review and observe specific health and safety requirements (e.g., personal protective equipment [PPE]) specified in the project-specific health and safety plan for this activity.

6.0 EQUIPMENT LIST

- Wood for decontamination pad construction, when applicable (see Section 7.1).

- Tools for constructing decontamination pad frame, when applicable (see Section 7.1).
- Visqueen sheeting or comparable material to cover decontamination pad frame, when applicable (see Section 7.1).
- Wash/drying racks for auger flights and drill/drive rods, when applicable (see Section 7.2).
- PPE as specified in the project health and safety plan.
- Soap and water for washing and rinsing.
- Deionized water for final rinsing.
- Solvents (e.g., pesticide-grade isopropanol) for rinsing (see applicable portions of Section 7.2).
- Tubs, buckets, etc. for containerizing rinse water (see applicable portions of Section 7.2).
- Sample bottles for collecting rinsate blanks (see Section 7.2).
- Calibrated photoionization detector (PID) or flame ionization detector (FID) to monitor decontaminated equipment for organic vapors gener ated through the existence of residual contamination or the presence of decontamination solvent remaining after the piece was rinsed.
- Aluminum foil or clear clean plastic bag for covering cleaned equipment (see applicable portions of Section 7.2).
- Paper towels or cloths for wiping.
- Brushes, scrapers, or other hand tools useful for removing solid materials from equipment.
- Clear plastic wrap for covering or wrapping la rge decontaminated equipment items (see Section 7.2.2).
- Drum-moving equipment for moving filled waste drums (optional) (see Section 7.3).
- Drum labels for waste drums (see Attachment A).

7.0 PROCEDURES

The process of decontamination is accomplished through the removal of contam inants, neutralization of contaminants, or isolation of contaminants. To accomplish this acti vity, preparation is required including site preparation, equipment sele ction, and evaluation of the decontamination requirements and processes. Site contaminant types, concentrations, and media types are primary drivers in the selection of the types of decontamination and where it will be conducted. For pur poses of this SOP, discussion is limited to decontamination procedures for general environmental investigations.

Decontamination processes will be performed at the lo cation(s) specified in project-specific planning documents. Typical decontamination locations include the following:

- Temporary decontamination pads/facilities
- Sample locations
- Centralized decontamination pad/facilities

- Combination of some or all of the above

The following discussion includes general considerations for the decontamination process. Specific construction and implementation procedures will be as specified in the project-specific planning documents and/or may be as dictated by site-specific conditions as long as the intent of the requirements in the planning documents is met. This intent is to contain any residual fluids and solids generated through the decontamination process.

7.1 Decontamination Pad Design/Construction Considerations

7.1.1 Temporary Decontamination Pads

Temporary decontamination pads may be constructed at satellite locations within the site area in support of temporary work areas. These structures are generally constructed to support the decontamination of heavy equipment such as drill rigs and earth-moving equipment but can be employed for smaller articles.

The purpose of the decontamination pad is to cont ain wash waters and potentially contaminated soil generated during decontamination procedures. Therefore, construction of these pads should take into account the following considerations:

- Site location – The decontamination site sele cted should be far enough from the work site to maximize decontamination effectiveness while mini mizing travel distance. The location of the decontamination site shall be selected to provide, in the judgment of the FOL or FOL designee, compliance with as many of the following characteristics as practicable:

  - Well removed from pedestrian/vehicle thoroughfares.
  - Avoidance of areas where control/custody cannot be maintained.
  - Avoidance of areas where potential releases of contaminated media or decontamination fluids may be compounded through access to storm water transport systems, streams, or other potentially sensitive areas.
  - Avoidance of potentially contaminated areas.
  - Avoidance of areas too close to the ongoing operation, where cross-contamination may occur.

  The selected decontamination site should include the following, where possible:

  - Areas where potable water and electricity are provided.

> ***Safety Reminder***
>
> When utilizing electrical power sources, either hard-wired or portable-generated sources, ensure that:
>
> - All power is routed through a Ground Fault Circuit Interrupter (GFCI).
>
> - All power cords are in good condition (no physical damage), rated for the intended energy load, and designated for outdoor use.
>
> In situations where accomplishing these elements is not possible, it will be necessary to implement a site electrical grounding program.

- Areas where support activities such as remo ving decontamination waters soil and sediment are possible without entering an active exclusion zone.

- Areas that offer sufficient size to carry out the specific decontamination sequence.

- Decontamination pad (decon pad) – The decon pad shall be constructed to meet the following characteristics:

  - Size – The size of the pad should be sufficient to accept the equipment to be decontaminated as well as permitting free movement around the equipment by the personnel conducting the decontamination. The size should permit these movements utilizing pressure/steam washer wands and hoses and minimizing splash due to work in close quarters.

  - Slope – An adequate slope will be constructed to permit the collection of water and potentially contaminated soil within a trough or sump constructed at one end. The collection point for wash waters should be of adequate distance that the decontamination workers do not have to walk through the wash waters while completing their tasks. Because the pad will be sloped, place a light coating of sand over the plastic to mini mize potential slips and falls. See the text about liners below.

  - Sidewalls – The sidewalls shall be at least 6 inches in height (or as high as possible if 6 inches is not achievable) to provide adequate containment for wash waters and soil. If splash represents a potential problem, splash guards should be construc ted to control overspray. Sidewalls may be constructed of wood, inflatables, sand bags, etc. to permit containment. Splash guards are typically wood frames with Visqueen coverings to control overspray.

  - Liner – Depending on the types of equipment and decontamination method to be used, the liner should be of sufficient thickness to prov ide a puncture-resistant barrier between the decontamination operation and the unprotected environment. Care should be taken to examine the surface area prior to placing the liner to re move sharp articles (sticks, stones, debris) that could puncture the liner. Liners are intended to form an impermeable barrier. The thickness may vary from a minimum recommended thickness of 10 mil to 30 mil. The desired thickness may be achieved through layering materials of lighter construction. It should be noted that various materials (rubber, polyethylene sheeting) become s lippery when wet. To minimize this potential hazard associated with a sloped liner, a light coat ing of sand shall be applied to provide traction as necessary.

  - Wash/drying racks – Auger flights, drill/drive rods, and similar equipment require racks positioned off of the ground to permit these articles to be washed, drained, and dried while secured from falling during this process.

For decontamination of direct-push technology (DPT) equipment, the pad may be as simple as a mortar tub containing buckets of soapy water for washing and an empty bucket to capture rinse waters. Decontamination may be conducted at the rear of the rig to permit rapid tool exchange.

- Maintenance – Maintain the decontamination area by:

  - Periodically clearing the work area of standing wa ter, soil, and debris, and coiling hoses to aid in eliminating slip, trip, and fall hazards. In additi on, these articles will reduce potential backsplash and cross-contamination.

- Regularly changing the decontamination fluids to ensure proper cleaning and prevent cross-contamination.

- PPE – Periodically evaluate the condition of, and maintain the decontamination equipment, including regular cleaning of face shields and safety glasses. This is critical to ensuring the safety of decontamination personnel and the integrity of the decontamination process, and it will ensure that equipment is functioning properly.

7.1.2 Decontamination Activities at Drill Rigs/DPT Units

During subsurface sampling activities including drilling and DPT activities, decontamination of drive rods, Macro Core Samplers, split spoons, etc. is typically conducted at an area adjacent to the operation. Decontamination is generally accomplished using a soap/water wash and rinse utilizing buckets and brushes. This area requires sufficient preparation to accomplish the decontamination objectives.

Buckets shall be placed within mortar tubs or similar secondary containment tubs to prevent splash and spills from reaching unprotected environmental media. Drying racks shall be employed as directed for temporary pads to permit parts to dry and be evaluated prior to use/reuse. Methodology regarding this activity is provided in Section 7.2.

7.1.3 Decontamination Activities at Remote Sample Locations

When sampling at remote locations, sampling equipment such as trowels and pumps/tubing should be evacuated of potentially contaminated media to the extent possible. This equipment should be wrapped in plastic for transport to the temporary/centralized decontamination location for final cleaning and disposition. Flushing and cleaning of single-use equipment such as disposable trowels, tubing, and surgeon's gloves may allow disposal of this equipment after visible soil and water remnants have been removed.

7.2 Equipment Decontamination Procedures

The following represents procedures to be employed for the decontamination of equipment that may have contacted and/or accumulated contamination through site investigation activities.

7.2.1 Monitoring Well Sampling Equipment

7.2.1.1 Groundwater sampling equipment – This includes pumps inserted into monitoring wells such as bladder pumps, Whale pumps, and Redi-Flo pumps and reusable bailers, etc.

1. Evacuate to the extent possible, any purge water within the pump/bailer.

2. Scrub using soap and water and/or steam clean the outside of the pump/bailer and, if applicable, the pump tubing.

3. Insert the pump and tubing/bailer into a clean container of soapy water. Pump/run a sufficient amount of soapy water through the pump/bailer to flush out any residual well water. After the pump is flushed, circulate soapy water through the pump to ensure that the internal components are thoroughly flushed.

4. Remove the pump and tubing/bailer from the container

5. Rinse external pump components using tap water.

6. Insert the pump and tubing/bailer into a clean container of tap water. Pump/run a sufficient amount of tap water through the pump/bailer to evacuate all of the soapy water (until clear).

CAUTION

Do not rinse PE, PVC, and associated tubing with solvents –
Use the procedures defined in the project-specific planning documents. If they are not defined, contact the FOL for guidance. The solvent rinse described in Step 7 may be omitted if groundwater does not contain oil, grease, PAHs, PCBs, or other hard to remove organic materials.

7. If groundwater contains or is suspected to contain oil, grease, PAHs, PCBs, or other hard to remove organic materials, rinse the equipment to be cleaned with pesticide-grade isopropanol.

8. Pass deionized water through the hose to flush out the tap water and solvent residue as applicable.

9. Drain residual deionized water to the extent possible.

10. Allow components of the equipment to air dry.

11. For bladder pumps, disassemble the pump and wash the internal components with soap and water, then rinse with tap water, isopropanol, and deionized water and allow to dry. After the parts are dry, conduct a visual inspection and a monitoring instrument scan to ensure that potential contaminants and all decontamination solvent have been removed. Collect a rinsate blank in accordance with the project-specific planning documents to ensure that the decontamination process is functioning as intended. The typical frequency of collection for rinsate blanks is 1 per 20 field samples. In addition, wipe samples or field tests such as UV light may be used.

12. Wrap pump/bailer in aluminum foil or a clear clean plastic bag for storage.

SAFETY REMINDER

Remember when handling powered equipment to disconnect the power source and render the equipment to a zero energy state (both potential and kinetic) before opening valves, disconnecting lines, etc.

7.2.1.2 Electronic Water Level Indicators/Sounders/Tapes

During water level measurements, rinsing the extracted tape and probe with deionized water and wiping the surface of the extracted tape between locations is acceptable. However, periodic full decontamination should be conducted as follows:

1. Wash with soap and water
2. Rinse with tap water
3. Rinse with deionized water

NOTE

In situations where oil, grease, free product, other hard to remove materials are encountered, probes and exposed tapes should be washed in hot soapy water. If probes or tapes cannot be satisfactorily decontaminated (they are still stained, discolored, etc.), they should be removed from service.

7.2.1.3 Miscellaneous Equipment

Miscellaneous equipment including analytical equipment (water quality testing equipment) shall be cleaned per manufacturers' instructions. This generally includes wiping the sensor housing and rinsing with tap and deionized water.

Coolers/shipping containers employed to ship samples are received from the laboratory in a variety of conditions including marginal to extremely poor. Coolers shall be evaluated prior to use for the following:

- Structural integrity – Coolers missing handles or having breaks in the outer housing should be removed and not used. Notify the laboratory that the risk of shipping samples in the cooler(s) provided is too great and request a replacement unit.

- Cleanliness – As per protocol, only volatile organic samples are accompanied by a trip blank. If a cooler's cleanliness is in question (visibly dirty/stained) or if there are noticeable odors, the cooler should be decontaminated prior to use as follows:

  1. Wash with soap and water
  2. Rinse with tap water
  3. Dry

If these measures fail to clean the cooler to an acceptable level, remove the unit from use as a shipping container and ask the cooler provider (e.g., the analytical laboratory) to provide a replacement unit.

7.2.2 Downhole Drilling Equipment

This includes any portion of the drill rig that is over the borehole, including auger flights, drill stems, rods, and associated tooling that would extend over the borehole. The following procedure is to be employed prior to initiating the drilling/sampling activity, then between locations:

> ***CAUTION***
> Exercise care when using scrapers to remove soil and debris from downhole drilling equipment. Inadvertent slips of scrapers have resulted in cuts, scrapes, and injured knuckles, so use scrapers carefully when removing soil from these items.

1. Remove loose soil using shovels, scrapers, etc.

2. Through a combination of scrubbing using soap and water and/or steam cleaning or pressure washing, remove visible dirt/soil from the equipment being decontaminated.

> ***CAUTION***
> In Step 3, do not rinse PE, PVC, and associated tubing with solvents. The appropriate procedures should be defined within the project-specific planning documents. If they are not defined, contact the FOL for guidance. The solvent rinse described in Step 4 may be omitted if groundwater does not contain oil, grease, PAHs, PCBs, or other hard to remove organic materials.

3. Rinse the equipment with tap water, where applicable (steam cleaning and pressure washing incorporate rinsing as part of the process).

4. If the equipment has directly or indirectly contacted contaminat ed sample media and is known or suspected of being contaminated with oil, grease, PAHs, PCBs, or other hard to remove organic materials, rinse equipment with pesticide-grade isopropanol

5. To the extent possible, allow components to air dry.

6. If the decontaminated equipment is to be used imm ediately after decontamination, screen it with a calibrated photoionization detector (PID)/flame i onization detector (FID) to ensure that all contaminants and possible decontamination solv ents (if they were used) have been adequately removed.

7. Wrap or cover equipment in clear plastic until it is time to be used.

SAFETY REMINDER

Even when equipment is disconnected from power sources, dangers such as the following may persist:

Falls - An auger flight standing on its end may fall and injure someone. Secure all loose articles to prevent heavy articles from falling onto people or equipment.

Burns - Steam cleaner water is heated to more than 212 °F and exhibits thermal energy that can cause burns. Prevent contact of skin with hot water or surfaces.

High water pressure - Pressure washer discharge can have 2,000 to 4,000 psi of water pressure. Water under this amount of pressure can rupture skin and other human tissues. Water at 4,000 psi exiting a 0° tip can be dangerous because of its relatively high cutting power. The exit velocity and cutting power of the water are reduced when exiting a 40° fan tip, but damage to soft tissues is still possible.

In general, follow the rules below to avoid injury, equipment damage, or incomplete decontamination:

1. Read the operating manual and follow the manuf acturers' recommended safety practices before operating pressure washers and steam cleaners.

2. Never point the pressure washer or steam cl eaner at another person or use to clean your boots or other parts of your body. Water lacerations and bur ns may appear to be minor at first but can be life threatening. Do not attempt to hold small parts in your hand while washing them with high-temperature or high-pressure water.

3. Always wear PPE as specified in the HASP such as:

   - Hard hat, safety glasses, splash shield, impermeable apron or splash suit, and hearing protection. Remember that excessive noise is a hazard when operating gas-powered engines and electrically driven pressure washers. PPE w ill be identified in your project specific planning documents.

4. Inspect each device before use. An inspection checklist will be pr ovided in the project-specific planning documents. If it is a rented device, safety m easures are typically provided by the vendor. In all cases, if you are not familiar with the oper ation of a pressure washer/steam cleaner, do not operate it until you obtain and thoroughly review operating instructions and recommended safety practices.

5. Do not modify equipment unless the manufacturer has approved the modifications.

7.2.3 Soil/Sediment Sampling Equipment

This section applies to soil sampling equipment including but not limited to hand augers, stainless steel trowels/spoons, bowls, dredges, scoops, split spoons, Macro Core samplers, etc.

1. Remove all loose soil from the equipment through manual means.

2. Through a combination of scrubbing using soap and water and/or steam cleaning or pressure washing, remove visible dirt/soil from the equipment.

3. Rinse the equipment with tap water.

> ***CAUTION***
> Do not rinse PE, PVC, and associated tubing with solvents. The appropriate procedures should be defined within the project-specific planning documents. If they are not defined, contact the FOL for guidance. The solvent rinse described in Step 4 may be omitted if groundwater does not contain oil, grease, PAHs, PCBs, or other hard to remove organic materials.

4. If the equipment is contaminated or suspected to be contaminated with oil, grease, PAHs, PCBs, or other hard to remove organic materials, rinse the equipment with pesticide-grade isopropanol.

5. Rinse the equipment with deionized water.

6. To the extent possible, allow components to air dry.

7. If the equipment is to be used immediately after decontamination, screen it with a calibrated PID/FID to ensure that all solvents (if they were used) and trace contaminants have been adequately removed.

8. After the equipment has dried, wrap it in aluminum foil for storage until use.

Dredges employed in sediment sampling are typically decontaminated as follows:

- Remove the sediment sample from the sampling device

- If sufficient associated surface water is available at the sampling site, place the dredge in the water and flush to remove visible sediment.

- Extract the dredge and wash it in soap and water per the project-specific planning documents.

> ***CAUTION***
> When handling dredges, the primary safety concern is trapping fingers or extremities in the larger dredge samplers within the jaws or pinch points of the mechanical jaws. Keep hands, fingers, and extremities away from these pinch and compression points. Either handle the device by the rope or preferably lock the jaws in place to control the potential for closing during maintenance and/or cleaning.

7.3 Contact Waste/Materials

During the course of field investigations, disposable/single-use equipment becomes contaminated. These items include tubing, trowels, PPE (gloves, overboots, splash suits, etc.), and broken sample containers.

With the exception of the broken glass, single-use articles should be cleaned (washed and rinsed) of visible materials and disposed as normal refuse. The exception to this rule is that extremely soiled materials that cannot be cleaned shall be containerized for disposal in accordance with applicable federal, state, and local regulations.

7.3.1 Investigation-Derived Wastes - Decontamination Wash Waters and Sediments

> ***NOTE***
> Requirements for waste storage may differ from one facility to the next. Facility-specific directions for waste storage areas will be provided in project-specific documents, or separate direction will be provided by the Project Manager.

1. Assume that all investigation-derived waste (IDW) generated from decontamination activities contains the hazardous chemicals associated with the site unless there are analytical or other data to the contrary. Waste solution volumes could vary from a few gallons to several hundred gallons in cases where large equipment required cleaning.

2. Where possible, use filtering systems to extend the use of water within a closed system wash unit to recycle water and to reduce possible waste amounts.

> ***NOTE***
> Containerized waste rinse solutions are best stored in 55-gallon drums (or equivalent containers) that can be sealed until ultimate disposal at an approved facility.

3. Label waste storage containers appropriately labeled (see Attachment A).

4. Ensure that the IDW storage area is configured to meet the following specifications to permit access to the containers and to conduct spill/leak monitoring, sampling, and extraction when the disposal route is determined:

   - Enclose areas accessible by the general public using construction fencing and signs.
   - Stored materials in 55-gallon drums on pallets with four (or fewer) drums per pallet.
   - Maintain the retaining bolt and label on the outside of storage containers where readily visible.
   - Provide at least 4 feet of room between each row of pallets to allow access to containers for sampling, drum removal, and spill response.
   - As directed in project-specific planning documents, maintain an IDW Inventory List and provide the list to the site Point of Contact at the termination of each shift.
   - Maintain spill response equipment at the IDW storage area in case it is required for immediate access.

- Where possible, use equipment for moving cont ainers. Where not possible, obtain help to manipulate containers.

CAUTION

Each container of water can weigh up to 490 pounds. Each 55-gallon drum of wet soil can weigh more than 750 pounds. Fill drums and temporary containers to 80 percent capacity to minimize spill and handling difficulties. Use drum carts to move filled drums.

See safe lifting techniques provided in Section 4.4 of the Tetra Tech NUS, Inc. Health and Safety Guidance Manual.

When placing drums, keep your fingers out of pinch and smash points such as between the drums. In some cases such as well development and/or purge water, you can place the drums to be filled on the pallet and transport materials in smaller easier to handle containers.

7.4 Decontamination Evaluation

Upon decontamination of equipment, determine the effectiveness of the decontamination process in the following manner:

- Visual evaluation – A visual evaluation will be conducted to ensure the removal of particulate matter. This shall be done to ensure that the washing/rinsing process is working as intended.

- Instrument Screening – A properly calibrated PID/FID should be used to evaluate the presence of site contaminants and solvents used in the cleaning process. The air intake of the instrument shall be passed over the article to be evaluated. Avoid placing the instrument probe into residual waters. A PID/FID reading greater than the daily established background level requires a repeat of the decontamination process, followed by rescreening with the PID/FID. This sequence must be repeated until no instrument readings greater than the daily established background level are observed. It should be noted that the instrument scan is only viable if the contaminants are detectable within the instrument's capabilities.

NOTE

When required by project-specific planning documents, collection of rinsate blanks (see next step) shall be completed without exception unless approval to not collect these samples is obtained from the Project Manager.

- Collection of Rinsate Blanks – It is recommended that rinsate samples be collected to:

  - Evaluate the decontamination procedure representing different equipment applications (pumps versus drilling equipment) and different decontamination applications.

  - Single-use disposable equipment – The number of samples should represent different types of equipment as well as different lot numbers of single-use articles.

  - The collection and the frequency of collection of rinsate samples are as follows unless specified differently in the project-specific planning documents:

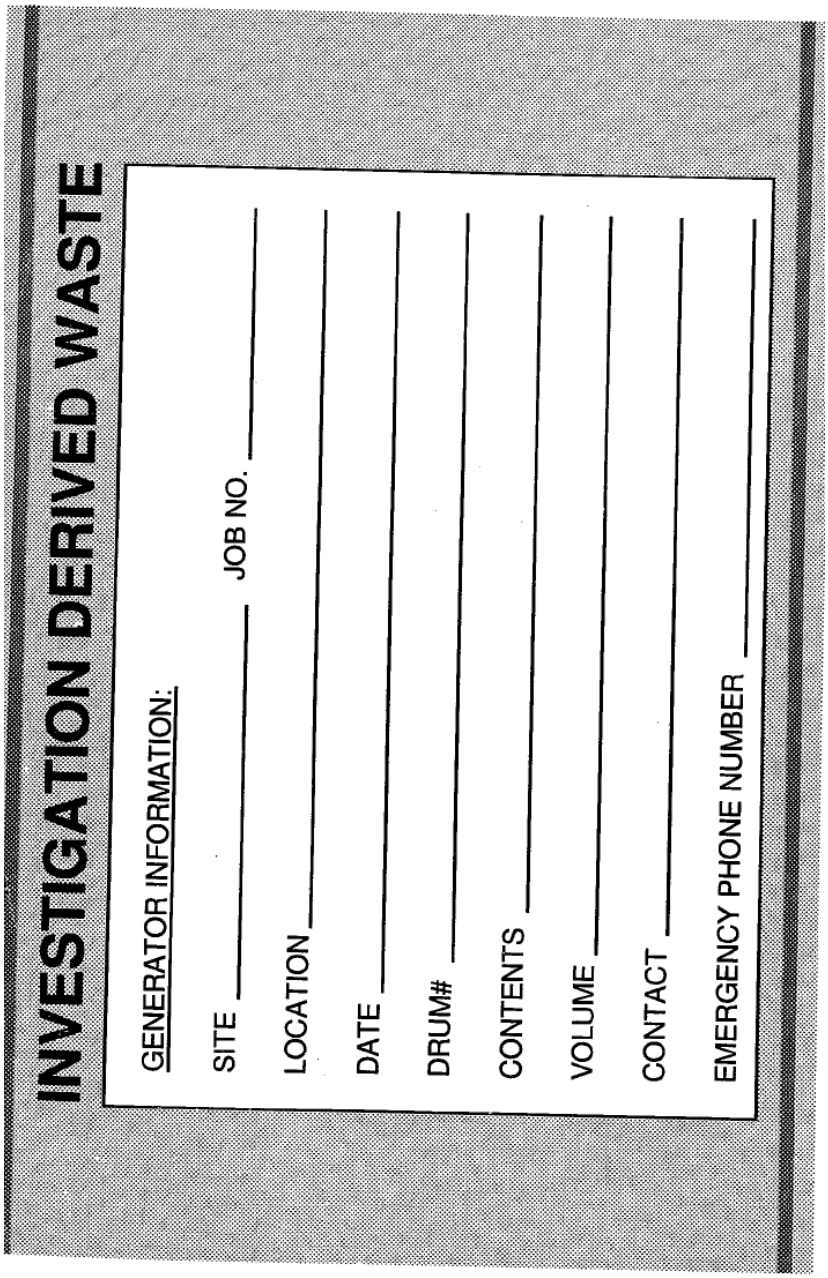
    - Per decontamination method
    - Per disposable article/batch number of disposable articles

NOTE

It is recommended that an initial rinsate sample be collected early in the project to ensure that the decontamination process is functioning properly and to avoid using a contaminated batch of single-use articles. It is recommended that a follow-up sample be collected later during the execution of the project to ensure that those conditions do not change.

Rinsate samples collection may be driven by types of and/or levels of contaminant. Difficult to remove contaminants, oils/greases, some PAHs/PCBs, etc. may also support the collection of additional rinsates due to the obvious challenges to the decontamination process. This is a field consideration to be determined by the FOL.

Attachment A
iDW Label



FM 1000. FIELD PLANNING AND MOBILIZATION

This SOP is advisory; however, the following procedures are designed as best management practices, for use as guidance for designing and implementing a field sampling program and when selecting a laboratory.

FM 2000. LABORATORY SCHEDULING

FM 2100. Selecting a Laboratory

1. CONSUMER RESPONSIBILITIES

Each organization that uses laboratory services has certain responsibilities to ensure that the laboratory has the appropriate credentials and that the data are useable for the intended needs, and acceptable to DEP. A consumer's responsibilities include:

1.1. Evaluating the Laboratory

1.1.1. Ensure that the laboratory has the proper credentials.

1.1.2. Ensure that the laboratory can produce data of a quality that will be acceptable to DEP.

1.2. Thinking in Terms of Quality not Dollars: A laboratory that produces data that are not acceptable to DEP usually means that the laboratory will need to repeat the work. It is more cost effective to select a laboratory that will meet the quality needs of the project even if that laboratory is not the lowest bidder.

1.3. Continuing Evaluation: In order to ensure that the laboratory provides data of a consistent quality, do not rely on just the initial evaluation of a laboratory. Other quality control measures will provide the ability to continuously evaluate the laboratory data quality.

1.4. Evaluating the Reported Data: Review the final laboratory reports against the original expectations and acceptable quality control measures.

1.5. Asking Questions: The consumer has the right to question laboratory results and receive a logical and clear response.

An informed client increases the probability of quality data and data acceptability.

FM 2110. IDENTIFYING LABORATORY NEEDS

The consumer should be able to identify these critical needs before considering any laboratory:

1. The purpose for which the data are needed.

   1.1. The consumer must determine DEP's expectations for data quality in terms of the precision, accuracy, and detection limit (reporting level or criteria) for each reported value.

   1.2. Examples include: permit compliance at some specified concentration levels; compliance monitoring at specified reporting levels; and site cleanup to specified soil and water criteria levels.

2. The benefits of using contracted or in-house analytical services.

3. The specific laboratory services that are required:

3.1. Are sample collection and sample analysis required, or just sample analysis.

3.2. Types of samples (groundwater, drinking water, soils, sediments, hazardous wastes, etc.).

3.3. The sample delivery schedule including:

3.3.1. The number of samples to be collected.

3.3.2. The frequency with which samples will be submitted to the laboratory.

3.3.3. The types of matrices to be analyzed.

3.4. The test methods that must be used (normally found in the permit requirements, consent orders, contracts, or relevant rules).

3.5. The expected quality based on DEP's requirements.

3.6. The expected turnaround time for laboratory analysis.

3.7. The deliverables including the report format.

3.8. Field related services such as:

3.8.1. Sample collection

3.8.2. Sample containers

3.8.3. Sample preservation

3.8.4. Equipment rental or cleaning services; or

3.8.5. Instrument calibration services.

4. Any required laboratory credentials such as certification.

5. Identifying key personnel in the consumer's organization that will be interfacing with the laboratory:

5.1. Administrative contact: Usually responsible for obtaining laboratory services.

5.2. Technical contact: Usually a person who will be evaluating the laboratory's performance.

5.3. Sample control contact: Usually a person who will be scheduling services with the laboratory.

6. Have an understanding of the current market price for the tests to be performed.

6.1. Gather information on pricing from several laboratories.

6.2. Request current and historical pricing schedules.

FM 2120. EVALUATING THE LABORATORY

1. LABORATORY CREDENTIALS

1.1. The laboratory must hold National Environmental Laboratory Accreditation Program (NELAP) certification from the Florida Department of Health's Environmental Laboratory Certification Program (DoH ELCP).

1.2. Out-of-state laboratories must be either certified by DoH, or be NELAP-certified by another state **with secondary accreditation** by DoH.

1.3. The laboratory must be certified for the test technology, analyte, and matrices that will be requested. This does not apply to analysis being done for drinking water.

1.4. Request a copy of the Current Certification and Analyte Sheets (must be for the current fiscal year which runs July 1 to June 30).

1.5. Verify the certification through the DEP Web Site, or the DoH offices.

2. ON-SITE VISIT

Conduct an on-site visit to verify the laboratory's capabilities and to determine if the laboratory has the equipment and personnel resources necessary for proposed services.

2.1. The laboratory must show a willingness to meet the client's needs.

2.2. The laboratory (both the analytical and administrative areas) should appear organized.

2.3. The analytical staff must be knowledgeable about the services to be provided.

2.3.1. At least one person (supervisor or analyst) must be experienced in performing all activities on the proposed scope of work.

2.4. The administrative staff must appear organized.

2.5. The laboratory must have the capacity to accommodate the proposed scope of work in terms of personnel and equipment.

3. LABORATORY PERFORMANCE EVALUATION

3.1. Blind Check Samples: Prior to contract signing or any agreement, submit a set of blind check samples to the laboratory.

3.1.1. A blind check sample is a sample in a real matrix (water, soil, sediment, etc.) that appears to be a real sample, except that the submitter has a list of the components and their known concentration values.

3.1.2. Submit the sample(s) to the laboratory as a routine sample(s).

3.1.3. Evaluate the results of the reported values against the certified values in the sample(s).

3.1.4. The values must be within the laboratory's stated precision for the measurement.

4. CUSTOMER SATISFACTION

4.1. Obtain a list of current and previous clients.

4.2. Call several of the clients to determine:

- Satisfaction with laboratory
- Were problems resolved satisfactorily?
- Reasons for not using the laboratory (if applicable)
- Reasons for using the laboratory

5. FISCAL STABILITY

5.1. Request a copy of the current financial statement.

FM 2130. CONTRACTING

1. PURPOSE

   1.1. Provide a detailed list of the scope of services to be contracted.

   1.2. Include the purpose for which the data are to be used (permit, compliance, etc.).

2. KEY CONTACTS: Identify key contacts for both laboratory and client:

   2.1. Administrative: Dealing with billing, contract writing, invoicing, etc.

   2.2. Technical: Dealing with data, and quality control issues and problems.

   2.3. Sample Control: Dealing with scheduling, shipping supplies, sample receipt.

3. ANTICIPATED NEEDS: Specify:

   3.1. The schedule of activities;

   3.2. The expected number of samples, analytes, matrices and tests; and

   3.3. Field support services, including containers, preservatives, cleaning and calibration services.

4. EXPECTATIONS

   4.1. Certification

      4.1.1. The laboratory must maintain certification for the analyte, technology, and matrices to be performed.

      4.1.2. The laboratory must immediately notify its clients if the certification status for any analyte changes.

      4.1.3. The laboratory must state that is will generate all results in strict compliance with the National Environmental Laboratory Accreditation Conference (NELAC) Standards.

      4.1.4. The laboratory must flag and justify any results that were not generated in accordance with NELAC.

   4.2. Analytical Expectations

      4.2.1. Provide a list of analytical methods to be performed and the matrices for each method.

      4.2.2. Provide a copy of the permit, QAPP, Sampling Plan or other document that outlines DEP's requirements.

      4.2.3. Specify the expected turn-around time for the analyses.

      4.2.4. Specify the shipping schedule if sample containers or supplies are to be provided.

   4.3. Container/Equipment Services: State the scope of container and equipment services:

      4.3.1. Precleaned Containers: Types and Numbers

         4.3.1.1. Must be cleaned according to DEP SOP procedures (see FC 1000) or purchased precleaned from a vendor.

         4.3.1.2. Provide copy of procedures, if the laboratory does not follow the DEP SOP procedures.

4.3.1.3. Determine if containers must be certified clean by either the laboratory or the vendor.

4.3.2. Preservatives

4.3.2.1. Premeasured into containers, where appropriate.

4.3.2.2. Provided in appropriate containers with dispensing implement.

4.3.3. Equipment

4.3.3.1. Type and numbers.

4.3.3.2. Condition of equipment (precleaned, etc.).

4.3.3.3. Equipment must be cleaned according to DEP SOP procedures (see FC 1000). Obtain a copy of the laboratory procedures if the laboratory does not follow the DEP SOP procedures.

4.3.3.4. Determine if equipment must be certified clean by the laboratory.

4.3.4. Equipment Calibration

4.3.4.1. The calibration method;

4.3.4.2. The frequency of calibration;

4.3.4.3. Preventative maintenance on instrument;

4.3.4.4. Certification statement verifying the calibration; and

4.3.4.5. Documentation of all maintenance and calibrations in laboratory records.

4.4. Quality Control

4.4.1. State adherence to NELAC quality control requirements.

4.4.2. Specify any additional quality control measures that are required but are different from NELAC.

4.4.3. Specify acceptable ranges for spikes, duplicates, surrogates, and other QC measures if appropriate.

4.5. Custody/Sample Tracking

4.5.1. Specify adherence to NELAP documentation and record keeping requirements.

4.5.2. State a time-period for retaining all records if greater than 5 years.

4.5.3. Make arrangement for transfer of records should the laboratory go out of business or transfer ownership before the records retention time period has lapsed.

4.5.4. Specify the level of custody (routine, legal, etc.).

4.6. Minimum Reporting Levels

4.6.1. Provide the laboratory with the minimum acceptable values to be reported (method detection limit, etc.).

4.6.2. Describe contingencies if these levels cannot be met.

4.7. Reporting Format

4.7.1. All analytical reports issued by the laboratory must comply with DEP and NELAP reporting requirements.

4.7.2. Specify whether the information must be provided as hardcopy, electronic or both.

4.7.2.1. If electronic, specify the format for submission.

4.7.3. The use of appropriate DEP data qualifiers (see Table FM 1000-1) must be used.

4.8. Deliverables: In addition to the NELAP-compliant report, specify any other deliverables that must be provided with the laboratory report such as:

- Laboratory Quality Control results;
- Field Quality Control results;
- Performance Test results;
- Copies of all raw data and associated records;
- Written narrative of the analytical event; and/or
- Description of any modifications to methods.

4.9. Subcontracting

4.9.1. The laboratory must inform the client **before** any analytical services are subcontracted to another laboratory.

4.9.2. The laboratory must ensure that the subcontracted laboratory meets the same qualifications and requirements as the primary laboratory.

4.9.3. If the results from subcontracted laboratories are incorporated into the final laboratory report, the subcontracted results must be clearly identified.

4.10. Method Modifications

4.10.1. The laboratory must identify any modifications that have been made to the requested analytical methods.

4.10.2. The client must be notified of any method modifications prior to use in the laboratory, and must provide written consent.

4.11. Dilutions

4.11.1. Negotiate how multiple dilutions will be handled. They may be considered a separate analysis and therefore an additional cost.

4.11.2. Agree to pay for the analysis of dilutions only if:

4.11.2.1. The sample concentration exceeds the calibration range and the laboratory was not aware of the expected sample concentration; or

4.11.2.2. A dilution is required to quantitate all required components.

5. PENALTIES AND CONSEQUENCES

5.1. Negotiate penalties or other consequences (no payment) for these problems:

- Failure to provide data or associated (expected) information;
- Failure to meet deadlines;
- Failure to provide acceptable data; and
- Failure to meet contract requirements.

5.2. Consider these consequences:

- Costs of resampling;
- Fines incurred because of unacceptable data;
- Costs associated with having evaluated and/or processed unacceptable data; and
- Reanalysis costs (if reanalysis is due to laboratory error or failed QC).

5.3. Reserve the right to reject data. If any data are used, laboratory should be paid according to negotiated terms.

FM 2140. ON-GOING EVALUATION

1. Monitor laboratory's performance against the specific contract requirements.
2. Continue to use blind QC samples as a measure of routine performance.
   2.1. Vendor supplied samples;
   2.2. Samples prepared to a known concentration; or
   2.3. Split samples with another laboratory.

FM 2150. DATA REVIEW

1. Review the data for logical trends:
   1.1. Are the reported concentrations different from the routine (expected) levels?
   1.2. Is the same value reported for the same analyte (except non detects) in the same set of samples or over a historical period of time?
   1.3. Do the parts add up to the total?
      1.3.1. Ortho phosphate must be less than total phosphate.
      1.3.2. Total nitrate-nitrite must be equal to nitrate plus nitrite.
      1.3.3. Total values must be greater than or equal to dissolved values.
   1.4. Are different but related analyses consistent?
      1.4.1. High turbidity and high total suspended solids.
      1.4.2. High turbidity and increased method detection limits for other tests.
   1.5. Do results indicate a sample collection problem?
      1.5.1. High dissolved oxygen in groundwater.
      1.5.2. High turbidity and elevated metals results.
   1.6. Are the QC check samples within acceptable ranges?
      1.6.1. Are the ranges reasonable?
   1.7. Are non-detects reported correctly (should be a value with a "U")?
   1.8. Over the history of laboratory use, were any QC problems reported?
   1.9. Is there any laboratory or field blank contamination?

1.10. Do the reports contain all required information?

FM 2160. ASK QUESTIONS

Ask questions if:

- There are problems associated with the data review.
- The QC check sample data are not acceptable.
- The laboratory consistently reports the same QC failure.
- The laboratory uses different methods than requested.
- The laboratory subcontracts analyses without notifying the client.
- The laboratory does not meet contract requirements.
- The laboratory misses holding times.
- The laboratory fails to provide requested resource(s) (containers, calibration, etc.) in a timely manner.
- There any doubts about the acceptability of the data.
- Detection limits are above the expected values and the laboratory provides no reasonable explanation.

FM 2200. Scheduling Services

1. Notify the laboratory about the analytical and equipment needs at least a week in advance of the actual sampling trip.

2. Even if the trip is routine (monthly, weekly, quarterly compliance sampling), provide the laboratory with a written request. Include:

- Number and types of samples to be collected;
- Test methods to be performed;
- Expectations for quality control acceptance criteria (if not already listed in a contract);
- Estimated numbers of each type of container;
- Required preservatives, including whether the laboratory will dispense premeasured quantities into the sample containers;
- Preservation supplies such as graduated, disposable pipets;
- Additional preservatives (even if the containers are prepreserved);
- Sampling equipment including material construction;
- Shipping containers;
- Forms (both courier and transmittal/custody forms);
- Any calibration services;
- Estimated time of delivery;
- Expected turn-around time;

- Special needs such as "requires legal chain of custody" or "requires 24-hour turn-around time";
- Data processing services (such as completing regulatory forms); and
- Expected contamination levels. This is important if a highly contaminated site is sampled.

FM 3000. TRIP PLANNING

1. Ensure that everyone involved with the event understands the purpose of the trip:

   1.1. Review the associated sampling plan, quality assurance project plan or permit requirements.

   1.2. Review the applicable safety plans and site files.

2. Determine the number of people that will be required to complete the sampling activities within the allotted time frame. For safety and efficiency, a field team should consist of at least two people.

3. Identify sampling team member(s) and schedule a meeting of the sampling team.

   3.1. Develop a detailed itinerary and schedule.

      3.1.1. Plan to sample from the least contaminated to the most contaminated sampling point.

      3.1.2. Plan to work upstream in flowing water.

   3.2. Review personnel training and make assignments based on experience.

      3.2.1. Ensure that at least one trained, experienced individual is part of the team.

   3.3. Review the SOPs and any associated documents (sampling plan, quality assurance project plan, permit, etc.).

   3.4. Review project/site files for unusual procedures or site peculiarities.

   3.5. Review the safety plan and discuss contingencies (weather, broken equipment, site access, etc.).

      3.5.1. If the sampling event is more than 3 - 5 days, a written contingency plan is recommended.

      3.5.2. If a boat will be used, a float plan is highly recommended.

      3.5.3. At a minimum discuss and have available:

         3.5.3.1. Phone and directions to nearest emergency facility;

         3.5.3.2. Phone number(s) of supervisor and/or project manager;

         3.5.3.3. Locations of power lines and underground utilities; and

         3.5.3.4. Expected environmental hazards.

4. Schedule the date for deployment and the duration of the sampling event.

   4.1. Obtain the necessary entry permits, keys, etc.

   4.2. Identify name(s) and phone number(s) of landowner, tenant or other responsible party.

5. Assemble any needed maps, directions and site descriptions. Include information on:

   5.1. Traffic conditions and/or traffic patterns; and

   5.2. Parking areas.

6. Identify the number of sampling points, and for each sampling point:

   6.1. Determine the matrices that will be sampled;

   6.2. Identify the specific analyses to be performed per matrix;

   6.3. Identify the sampling equipment needs based on the matrix and analytes to be collected. Include tubing, mixing implements and other support equipment;

   6.4. Based on the analytical tests and the matrices, determine the number and types of sample containers;

   6.5. Based on the analytical tests and the matrices, determine the types of preservatives that will be needed;

   6.6. Determine what field measurements must be made; and

   6.7. Identify transportation mode to reach the location (boat, truck, etc.).

7. Calculate the total number of each container types (both preserved and unpreserved).

8. Determine the total number of sampling equipment sets (tubing, mixing trays, coring devices, etc.) that will be needed for the sampling event.

9. Notify the laboratory of the trip and arrange for necessary containers, preservatives and other supplies (see FM 2200).

10. Reserve appropriate vehicles.

11. Assemble all field records (notebooks, forms, transmittal forms, etc.).

FM 4000. EQUIPMENT AND SUPPLY PREPARATION

1. SAMPLING EQUIPMENT: Assemble all equipment identified in FM 3000, section 8.

   1.1. Inspect equipment for cracks, breaks, and other signs of wear.

   1.2. If necessary, repair any equipment and document the repairs in appropriate maintenance logs.

   1.3. Reclean any equipment that was cleaned but not protected from the environment (stored on dusty shelves).

      1.3.1. If not already clean, decontaminate equipment according to FC 1000.

      1.3.2. Clean all transport ice chests and water transport containers (see FC 1190 and FC 1180, respectively).

   1.4. Check to make sure fuel and battery powered pumps are working.

   1.5. See "Field Sample Collection Equipment Checklist".

2. FIELD MEASUREMENTS: Assemble field instruments to make the measurements identified in FM 3000, section 6.6.

   2.1. Inspect instruments for damage.

2.1.1. Repair and/or replace parts as necessary, and document in appropriate maintenance logs.

2.1.2. Assemble the appropriate calibration standards and supplies.

2.1.3. Determine the accuracy of the instruments by either performing an initial calibration or checking the calibration before leaving the base of operations. Document the calibration check.

2.2. See "General Field Support Equipment Checklist", item 7.

3. DOCUMENTATION: Assemble field record supplies:

- Notebooks, and/or forms
- Indelible/waterproof pens
- Clipboards
- Cameras
- GPS unit, if needed
- See "General Field Support Equipment Checklist ".

4. SAMPLE CONTAINERS: Assemble the appropriate types of sample containers or obtain them from the contracted laboratory. See "General Field Support Equipment Checklist", item 8.

5. PRESERVATIVES: Assemble preservation supplies if not provided by the laboratory.

5.1. Discard any old solutions; clean containers; and prepare fresh solutions.

5.2. See "General Field Support Equipment Checklist", item 2.

6. FIELD DECONTAMINATION SUPPLIES: Assemble field decontamination supplies.

6.1. Discard any old solutions; clean containers; and prepare fresh solutions.

6.2. Discard analyte-free water and obtain fresh water.

6.3. See "General Field Support Equipment Checklist", item 1.

7. SHIPPING SUPPLIES: Assemble shipping supplies:

7.1. Determine nearest point to obtain ice;

7.2. Marking pens, shipping labels, tape, custody seals (if required);

7.3. See "General Field Support Equipment Checklist", item 3.

8. VEHICLES:

8.1. Make sure vehicle maintenance is up-to-date.

8.2. Check fluids.

8.3. Check tire pressure.

8.4. Check fuel and fuel supply.

8.5. See "General Field Support Equipment Checklist", item 10.

9. SAFETY EQUIPMENT: Assemble any needed safety equipment:
    - Protective gloves.
    - Protective clothing including boots.
    - SCUBA gear or other supplied air supply.
    - First aid kit.
    - Drinking water.
    - Float plan.
    - Address and phone numbers for nearest emergency room.
    - See "General Field Support Equipment Checklist", item 6.

Appendix FM 1000

Tables, Figures and Checklists

Table FM 1000-1 Data Qualifier Codes

General Field Support Equipment Checklist

Field Sample Collection Equipment Checklist

Table FM 1000-1
DATA QUALIFIER CODES

The following codes shall be used by laboratories and/or field organizations when reporting data values that either meet the specified description outlined below or do not meet the quality control criteria of the laboratory:

Symbol	Meaning
A	Value reported is the arithmetic mean (average) of two or more determinations. This code shall be used if the reported value is the average of results for two or more discrete and separate samples. These samples shall have been processed and analyzed independently. Do not use this code if the data are the result of replicate analysis on the same sample aliquot, extract or digestate.
B	Results based upon colony counts outside the acceptable range. This code applies to microbiological tests and specifically to membrane filter colony counts. The code is to be used if the colony count is generated from a plate in which the total number of coliform colonies is outside the method indicated ideal range. This code is not to be used if a 100 mL sample has been filtered and the colony count is less than the lower value of the ideal range.
F	When reporting species: F indicates the female sex.
H	Value based on field kit determination; results may not be accurate. This code shall be used if a field screening test (i.e., field gas chromatograph data, immunoassay, vendor-supplied field kit, etc.) was used to generate the value and the field kit or method has not been recognized by the Department as equivalent to laboratory methods.
I	The reported value is greater than or equal to the laboratory method detection limit but less than the laboratory practical quantitation limit.
J	Estimated value. A “J” value shall be accompanied by a detailed explanation to justify the reason(s) for designating the value as estimated. Where possible, the organization shall report whether the actual value is estimated to be less than or greater than the reported value. A “J” value shall not be used as a substitute for K, L, M, T, V, or Y, however, if additional reasons exist for identifying the value as an estimate (e.g., matrix spiked failed to meet acceptance criteria), the “J” code may be added to a K, L, M, T, V, or Y. Examples of situations in which a “J” code must be reported include: instances where a quality control item associated with the reported value failed to meet the established quality control criteria (the specific failure must be identified); instances when the sample matrix interfered with the ability to make any accurate determination; instances when data are questionable because of improper laboratory or field protocols (e.g., composite sample was collected instead of a grab sample); instances when the analyte was detected at or above the method detection limit in a blank other than the method blank (such as calibration blank or field-generated blanks and the value of 10 times the blank value was equal to or greater than the associated sample value); or instances when the field or laboratory calibrations or calibration verifications did not meet calibration acceptance criteria.

Table FM 1000-1
DATA QUALIFIER CODES

Symbol	Meaning
K	Off-scale low. Actual value is known to be less than the value given. This code shall be used if:
	1. The value is less than the lowest calibration standard and the calibration curve is known to be non-linear; or
	2. The value is known to be less than the reported value based on sample size, dilution.
	This code shall not be used to report values that are less than the laboratory practical quantitation limit or laboratory method detection limit.
L	Off-scale high. Actual value is known to be greater than value given. To be used when the concentration of the analyte is above the acceptable level for quantitation (exceeds the linear range or highest calibration standard) and the calibration curve is known to exhibit a negative deflection.
M	When reporting chemical analyses: presence of material is verified but not quantified; the actual value is less than the value given. The reported value shall be the laboratory practical quantitation limit. This code shall be used if the level is too low to permit accurate quantification, but the estimated concentration is greater than or equal to the method detection limit. If the value is less than the method detection limit use “T” below.
N	Presumptive evidence of presence of material. This qualifier shall be used if:
	1. The component has been tentatively identified based on mass spectral library search; or
	2. There is an indication that the analyte is present, but quality control requirements for confirmation were not met (i.e., presence of analyte was not confirmed by alternative procedures).
O	Sampled, but analysis lost or not performed.
Q	Sample held beyond the accepted holding time. This code shall be used if the value is derived from a sample that was prepared or analyzed after the approved holding time restrictions for sample preparation or analysis.
T	Value reported is less than the laboratory method detection limit. The value is reported for informational purposes only and shall not be used in statistical analysis.
U	Indicates that the compound was analyzed for but not detected. This symbol shall be used to indicate that the specified component **was not** detected. The value associated with the qualifier shall be the laboratory method detection limit. Unless requested by the client, less than the method detection limit values shall not be reported (see “T” above).
V	Indicates that the analyte was detected at or above the method detection limit in both the sample and the associated method blank and the value of 10 times the blank value was equal to or greater than the associated sample value. Note:

Table FM 1000-1
DATA QUALIFIER CODES

Symbol	Meaning
	unless specified by the method, the value in the blank shall not be subtracted from associated samples.
X	Indicates, when reporting results from a Stream Condition Index Analysis (LT 7200 and FS 7420), that insufficient individuals were present in the sample to achieve a minimum of 280 organisms for identification (the method calls for two aliquots of 140-160 organisms), suggesting either extreme environmental stress or a sampling error.
Y	The laboratory analysis was from an improperly preserved sample. The data may not be accurate.
Z	Too many colonies were present for accurate counting. Historically, this condition has been reported as “too numerous to count” (TNTC). The “Z” qualifier code shall be reported when the total number of colonies of all types is more than 200 in all dilutions of the sample. When applicable to the observed test results, a numeric value for the colony count for the microorganism tested shall be estimated from the highest dilution factor (smallest sample volume) used for the test and reported with the qualifier code.
?	Data are rejected and should not be used. Some or all of the quality control data for the analyte were outside criteria, and the presence or absence of the analyte cannot be determined from the data.
*	Not reported due to interference.

The following codes deal with certain aspects of field activities. The codes shall be used if the laboratory has knowledge of the specific sampling event. The codes shall be added by the organization collecting samples if they apply:

Symbol	Meaning
D	Measurement was made in the field (i.e., in situ). This code applies to any value (except field measurements of pH, specific conductance, dissolved oxygen, temperature, total residual chlorine, transparency, turbidity or salinity) that was obtained under field conditions using approved analytical methods. If the parameter code specifies a field measurement (e.g., “Field pH”), this code is not required.
E	Indicates that extra samples were taken at composite stations.
R	Significant rain in the past 48 hours. (Significant rain typically involves rain in excess of 1/2 inch within the past 48 hours.) This code shall be used when the rainfall might contribute to a lower than normal value.
!	Data deviate from historically established concentration ranges.

General Field Support Equipment Checklist

Date: _____ Project/Site: _____

DECONTAMINATION SUPPLIES

- ☐ Basins, buckets or bowls to hold wash water and various rinse waters
- ☐ Brushes or other implements to clean equipment
- ☐ Detergents
  - ☐ Liqui-Nox or equivalent
  - ☐ Alconox or equivalent
- ☐ Acids
  - ☐ Nitric
  - ☐ Hydrochloric
- ☐ Solvents
- ☐ Pesticide grade isopropanol
  - ☐ Other: _____
- ☐ Protective wrapping
  - ☐ Foil
  - ☐ Untreated Plastic bags
  - ☐ Bubble wrap
- ☐ Analyte-free water
  - ☐ Distilled in HDPE
  - ☐ Deionized in HDPE
- ☐ Organic-free in HDPE, Teflon or glass
- ☐ Dispensing bottles
- ☐ HDPE for acids and detergents
- ☐ Teflon for solvents and organic-free water
- ☐ Paper towels or other absorbent material
- ☐ Containers for IDW

PRESERVATION SUPPLIES

- ☐ Acids
  - ☐ Nitric
  - ☐ Hydrochloric
  - ☐ Sulfuric
- ☐ Dechlorination reagents
  - ☐ Sodium thiosulfate
  - ☐ Ascorbic acid
- ☐ Sodium hydroxide
- ☐ Dispensing devices
- ☐ Graduated disposable plastic pipets
- ☐ Glass Pasteur pipets
- ☐ Bulbs
- ☐ Premeasured reagents in vials
- ☐ Narrow range pH paper (range of no more than 3 pH units)
  - ☐ pH range of 1 – 3
  - ☐ pH range of 11 – 14
  - ☐ pH range of 6 – 8
- ☐ Cyanide processing
  - ☐ Sulfide test paper
  - ☐ Precipitating Chemical
  - ☐ Cadmium nitrate or
  - ☐ Cadmium carbonate or
  - ☐ Lead nitrate or
  - ☐ Lead carbonate
  - ☐ KI starch paper
  - ☐ Ascorbic acid
  - ☐ Filter paper

SAMPLE TRANSPORTATION SUPPLIES

- ☐ Ice chests
- ☐ Wet ice
- ☐ Sealing tape
- ☐ Shipping labels
- ☐ Shipping forms
- ☐ Bubble wrap
- ☐ Plastic bags
- ☐ Vermiculite
- ☐ Custody seals

DOCUMENTATION SUPPLIES

- ☐ Notebooks/logs/field forms
- ☐ Pens and markers (waterproof)
- ☐ Sample container labels/tags
- ☐ Custody tags
- ☐ Custody/transmittal forms
- ☐ Clipboard
- ☐ Camera
  - ☐ Film
- ☐ GPS equipment
- ☐ Calculator

REFERENCE MATERIALS

- ☐ Site maps and directions
- ☐ QAPP
- ☐ Sampling plan
- ☐ SOPs
- ☐ Itinerary
- ☐ Float plan
- ☐ Contingency plan

HEALTH & SAFETY SUPPLIES

- ☐ Cell phone
- ☐ First aid kit
- ☐ Drinking water
- ☐ Protective gloves
- ☐ Insect repellent
- ☐ Sunscreen
- ☐ Numbers for nearest emergency facilities
- ☐ Safety goggles
- ☐ Applicable MSDS sheets
- ☐ Respirators
- ☐ Fire extinguisher
- ☐ Hard hats
- ☐ Flotation jackets
- ☐ Cable cutters
- ☐ Traffic cones
- ☐ SCUBA gear
- ☐ SCBA gear
- ☐ Other personal protection gear

FIELD MEASUREMENT EQUIPMENT

- ☐ Lint-free tissues
- ☐ Flow-through cells
- ☐ pH meter
  - ☐ 4, 7 & 10 buffers
- ☐ Conductivity meter
  - ☐ Solution at expected conductivity
- ☐ DO meter
- ☐ Turbidimeter
  - ☐ Gel or Formazin standards

General Field Support Equipment Checklist

Date: _____ Project/Site: _____

- ☐ Residual chlorine
  - ☐ Secondary or primary standards
- ☐ Secchi disk
- ☐ MultiProbe

SAMPLE CONTAINERS

- ☐ Extractable Organics
- ☐ Volatile Organics
- ☐ Nutrients
  - ☐ Glass
  - ☐ Plastic
- ☐ Inorganic Non-metallics
  - ☐ Glass
  - ☐ Plastic
- ☐ Physical Parameters
  - ☐ Glass
  - ☐ Plastic
- ☐ Metals
  - ☐ Glass
  - ☐ Plastic
- ☐ Microbiology
  - ☐ Glass
  - ☐ Plastic
- ☐ Whole Effluent Toxicity
- ☐ Tissues
- ☐ Macrobenthic invertebrates
- ☐ Periphyton
- ☐ Sediment/Soil volatiles
- ☐ Sediment/Soil

Remember:

- ☐ Extra containers
- ☐ Extra VOC septa

FILTRATION EQUIPMENT

- ☐ 1 µm filter units
- ☐ 0.45 µm filters
- ☐ Peristaltic pump
- ☐ Pressurized bailers
- ☐ Syringe with Luer-Lok fitting
- ☐ Tripod filter with pressure/vacuum source
- ☐ Forceps for handling filters

VEHICLES

- ☐ Truck
- ☐ Fuel
- ☐ Boat
- ☐ Fuel
- ☐ Motor
- ☐ Paddles/oars
- ☐ Safety vests

MISCELLANEOUS SUPPLIES

- ☐ Hip boots
- ☐ Chest waders
- ☐ Rain gear
- ☐ Tool kit
- ☐ Extra batteries
- ☐ Stopwatch

Field Sample Collection Equipment Checklist

Date: _____ Project/Site: _____

GROUNDWATER

Pumps
- ☐ Peristaltic
- ☐ Centrifugal
- ☐ Variable speed submersible
- ☐ Submersible
- ☐ Variable speed bladder
- ☐ Bladder

Tubing
- ☐ Teflon ___Sets
- ☐ Polyethylene ___Sets
- ☐ Polypropylene ___Sets
- ☐ Vinyl ___Sets
- ☐ Rubber ___Sets
- ☐ Tygon ___Sets

Bailers
- ☐ Teflon
- ☐ Stainless steel
- ☐ Polyethylene
- ☐ Acrylic
- ☐ PVC

Support Equipment
- ☐ Graduated containers for measuring purge water
- ☐ Containers for holding purge waters
- ☐ Water level measuring device
- ☐ Plastic sheeting
- ☐ Lanyard material
- ☐ Reels
- ☐ Energy source for pumps

SURFACE WATER

Pumps:
- ☐ Peristaltic
- ☐ Automatic composite sampler
- ☐ Other

Tubing
- ☐ Teflon™ ___Sets
- ☐ Polyethylene ___Sets
- ☐ Polypropylene ___Sets
- ☐ Vinyl ___Sets
- ☐ Rubber ___Sets
- ☐ Tygon ___Sets

Bailers
- ☐ Teflon
- ☐ Stainless Steel
- ☐ Polyethylene
- ☐ Acrylic
- ☐ PVC

Grab Sampling Devices:
- ☐ Dipper
- ☐ Kemmerer
- ☐ Alpha water sampler
- ☐ Niskin
- ☐ Beta sampler
- ☐ Retrieval lines

Mixing Implements
- ☐ Churn splitter

WASTEWATER
- ☐ Pond sampler
- ☐ Dippers
- ☐ Peristaltic pump

Tubing
- ☐ Teflon ___Sets
- ☐ Polyethylene ___Sets
- ☐ Polypropylene ___Sets
- ☐ Vinyl ___Sets
- ☐ Rubber ___Sets
- ☐ Tygon ___Sets
- ☐ Kemmerer
- ☐ Van Dorn
- ☐ Nansen
- ☐ Alpha bottle
- ☐ Beta bottle
- ☐ Niskin
- ☐ DO dunker
- ☐ Automatic composite sampler

Tubing
- ☐ Teflon ___Sets
- ☐ Polyethylene ___Sets
- ☐ Polypropylene ___Sets
- ☐ Vinyl ___Sets
- ☐ Rubber ___Sets
- ☐ Tygon ___Sets

Bailers
- ☐ Plastic
- ☐ Teflon
- ☐ Stainless steel

Scoops
- ☐ Plastic
- ☐ Teflon
- ☐ Stainless steel

Beakers
- ☐ Plastic
- ☐ Teflon
- ☐ Stainless steel

Buckets
- ☐ Plastic
- ☐ Stainless steel

SEDIMENTS

Dredges
- ☐ Petersen
- ☐ Ponar
- ☐ Ekman
- ☐ Young Grab
- ☐ Van Veen
- ☐ Shipek
- ☐ Orange-peel grab
- ☐ Smith-McIntyre grab
- ☐ Drag buckets
- ☐ Winch
- ☐ Cable/line
- ☐ Messenger

Coring Devices
- ☐ Stainless steel
- ☐ Glass
- ☐ Plastic
- ☐ Teflon-lined

SOIL
- ☐ Bucket auger
- ☐ Split spoon sampler
- ☐ Stainless steel shovel
- ☐ Garden shovel
- ☐ Stainless steel trowel or scoop
- ☐ Plastic trowel or scoop
- ☐ Trenching device
- ☐ Coring Devices
- ☐ Stainless steel
- ☐ Glass
- ☐ Plastic
- ☐ Teflon-lined
- ☐ Shelby tube
- ☐ EnCore

Field Sample Collection Equipment Checklist

Date: _____ Project/Site: _____

WASTE
- ☐ Stainless steel scoop
- ☐ Stainless steel spoons or spatulas
- ☐ Stainless steel push tubes
- ☐ Stainless steel auger
- ☐ Stainless steel Ponar dredge
- ☐ Glass coliwasa
- ☐ Drum thief
- ☐ Mucksucker
- ☐ Dipstick
- ☐ Stainless steel bacon bomb
- ☐ Stainless steel bailer
- ☐ Teflon bailer
- ☐ Peristaltic pump
- ☐ Stainless steel split spoon
- ☐ Roto-hammer
- ☐ Glass tubing

SHELLFISH
- ☐ Seine
- ☐ Trawl
- ☐ Bucket type/double pole
- ☐ Tong/Double handed grab
- ☐ Line or cable operated grab bucket
- ☐ Petersen
- ☐ Ponar
- ☐ Ekman
- ☐ Orange-peel grab
- ☐ Biological or hydraulic dredge
- ☐ Scoops/shovels
- ☐ Scrapers
- ☐ Rakes
- ☐ D-traps

Processing Equipment
- ☐ Holding trays
- ☐ Stainless steel shucking knife
- ☐ Calipers or ruler
- ☐ Aluminum foil
- ☐ Plastic bags

FINFISH
- ☐ Electrofishing devices
- ☐ Seines
- ☐ Trawls
- ☐ Angling
- ☐ Gill net
- ☐ Trammel net
- ☐ Hoop, fyke & pound nets
- ☐ D-traps

Processing Equipment
- ☐ Holding trays
- ☐ Measuring board or ruler
- ☐ Stainless steel descaler
- ☐ Stainless steel scalpel
- ☐ Balance
- ☐ Aluminum foil
- ☐ Plastic bags

BIOLOGICAL COMMUNITY SAMPLING

Phytoplankton
- ☐ Van Dorn
- ☐ Alpha bottle
- ☐ Logol's solution

Periphyton
- ☐ Periphytometer
- ☐ Microscope slides
- ☐ 100% buffered formalin
- ☐ Nylon twine

Qualitative Periphyton Sampling
- ☐ Stainless steel spatula/spool
- ☐ Stainless steel forceps
- ☐ Suction bulb
- ☐ Preservative
  - ☐ Buffered formalin
  - ☐ Lugol's solution
- ☐ M3
- ☐ Resealable plastic bags
- ☐ White picking pan

Benthic Macroinvertebrates
- ☐ Forceps
- ☐ Transfer pipettes
- ☐ White picking pans
- ☐ 10X hand lens
- ☐ Alcohol-filled jars
- ☐ Dip net (30 mesh)
- ☐ Hester-Dendy
- ☐ Coring device
- ☐ Dredge
  - ☐ Ekman
  - ☐ Petite ponar
- ☐ 30 mesh box sieve

FQ 1000. FIELD QUALITY CONTROL REQUIREMENTS

Field quality control measures monitor the sampling event to ensure that the collected samples are representative of the sample source.

Field-collected blanks must demonstrate that the collected samples have not been contaminated by:

- The sampling environment
- The sampling equipment
- The sample container
- The sampling preservatives
- Sample transport
- Sample storage

FQ 1100. Sample Containers

Sample containers must be free from contamination by the analytes of interest or any interfering constituents and must be compatible with the sample type.

FQ 1200. Sampling Operations

1. When collected, analyze all quality control samples for the same parameters as the associated samples.

1.1. When collected, collect blanks for the following parameter groups and tests:

- Volatile Organics
- Extractable Organics
- Metals
- Ultratrace Metals
- Inorganic Nonmetallics
- Radionuclides
- Petroleum Hydrocarbons and Oil & Grease
- Volatile Inorganics
- Aggregate Organics except Biochemical Oxygen Demand

1.2. Blanks are not required for:

- Microbiological (all types)
- Toxicity
- Field parameters such as pH, Specific Conductance, Residual Chlorine, Temperature, Light Penetration, Dissolved Oxygen, ORP and Salinity
- Radon

- Algal Growth Potential
- Biological Community
- Physical and Aggregate Properties
- Biochemical Oxygen Demand

2. Preserve, transport, document and handle all quality control samples as if they were samples. Once collected, they must remain with the sample set until the laboratory has received them.

3. Except for trip blanks, prepare all quality control samples **on-site in the field**.

3.1. Do not prepare precleaned equipment blanks in advance at the base of operations.

3.2. Do not prepare field-cleaned equipment blanks after leaving the sampling site.

4. Perform and document any field QC measures specified by the analytical method (such as trip blanks for volatile organics).

FQ 1210. QUALITY CONTROL BLANKS

FQ 1211. *Precleaned Equipment Blanks*

1. USE: Monitors on-site sampling environment, sampling equipment decontamination, sample container cleaning, the suitability of sample preservatives and analyte-free water, and sample transport and storage conditions for water, waste, soil, or sediment samples.

2. Collect these blanks using sampling equipment that has been brought to the site precleaned and ready for use. The cleaning procedures used for the blank collection must be identical to those used for the field sample collection.

3. Collect these blanks before the equipment set has been used.

4. Prepare equipment blanks by rinsing the sampling equipment set with the appropriate type of analyte-free water and collecting the rinse water in appropriate sample containers (see FQ 1100).

FQ 1212. *Field-Cleaned Equipment Blanks*

1. USE: Monitors on-site sampling environment, sampling equipment decontamination, sample container cleaning, the suitability of sample preservatives and analyte-free water, and sample transport and storage conditions.

2. Collect these blanks using sampling equipment that has been cleaned in the field (i.e., between sampling points). The cleaning procedures used for the blank collection must be identical to those used for the field sample collection.

3. Prepare field-cleaned equipment blanks immediately after the equipment is cleaned in the field and before leaving the sampling site.

4. Prepare equipment blanks by rinsing the sampling equipment set with the appropriate type of analyte-free water and collecting the rinse water in appropriate sample containers (see FQ 1100).

4.1. For intermediate sampling devices or equipment, site-water rinsing is defined as the decontamination step, if this is the only cleaning that will be performed on the equipment prior to collecting the sample.

4.1.1. In this case, collect the equipment blank after rinsing the intermediate device 3 times with site water

4.1.2. Follow the site-water rinses with 3 rinses using analyte-free water.

4.1.3. Collect the equipment blank with a subsequent rinse of the device using additional analyte-free water to collect sufficient blank volume.

FQ 1213. *Trip Blanks*

1. USE: Monitors sample container cleaning, the suitability of sample preservatives and analyte-free water, and sample transport and storage conditions.

2. The organization that is providing the VOC vials must provide the trip blanks by filling two or more VOC vials with analyte-free water and preservatives (if needed).

2.1. To prevent degradation of the trip blank, long-term storage of prepared trip blanks is not recommended.

3. These blanks are applicable if samples are to be analyzed for volatile constituents (volatile organics, methyl mercury, etc.) in water, waste, soils, or sediments.

4. Place a set of trip blanks in each transport container used to ship/store empty VOC vials. They must remain with the VOC vials during the sampling episode and must be transported to the analyzing laboratory in the same shipping or transport container(s) as the VOC samples.

5. Trip blanks must be opened **only** by the laboratory after the blank and associated samples have been received for analysis.

FQ 1214. *Field Blanks*

1. USE: Monitors on-site sampling environment, sample container cleaning, the suitability of sample preservatives and analyte-free water, and sample transport and storage conditions for water, waste, soil or sediment samples.

2. Prepare field blanks by pouring analyte-free water into sample containers for each parameter set to be collected.

3. Field blanks are not required if equipment blanks (FQ 1211 or FQ 1212) are collected.

FQ 1220. FIELD DUPLICATES

1. USE: Designed to measure the variability in the sampling process.

2. GENERAL CONSIDERATIONS:

2.1. Collect duplicates by **repeating** (simultaneously or in rapid succession) the entire sample acquisition technique that was used to obtain the first sample.

2.1.1. Collect, preserve, transport and document duplicates in the same manner as the samples. **These samples are not considered laboratory duplicates**.

2.2. When collected, analyze field duplicates for the same parameters as the associated samples.

2.3. If possible, collect duplicate samples from sampling locations where contamination is present.

2.4. Field duplicates must be collected if required by the analytical method and as required by a DEP program.

FQ 1221. *Water Duplicates*

Collect water duplicates by sampling from successively collected volumes (i.e., samples from the next volume of sample water).

FQ 1222. *Soil Duplicates*

Collect soil duplicates from the same sample source (i.e., soil from the same soil sampling device).

FQ 1230. MANDATORY FIELD QUALITY CONTROLS

1. The respondent, permittee or contractor and the sampling organization are responsible for ensuring that blanks (excluding trip blanks) are collected at a minimum of 5% of each reported test result/matrix combination for the life of a project.

1.1. Collect at least one blank for each reported test result/matrix combination each year for each project.

1.2. If a party wishes to claim that a positive result is due to external contamination sources during sample collection, transport or analysis, then at least one field collected blank (excludes trip blanks) must have been collected at the same time the samples were collected and analyzed with the same sample set.

1.3. A project will be defined by the organization responsible for collecting the samples for the project.

1.3.1. When applicable, define the scope of the project in conjunction with the appropriate DEP authority.

2. When collecting a set of blanks, use the following criteria:

2.1. Equipment Blanks:

2.1.1. Collect field-cleaned equipment blanks if any sample equipment decontamination is performed in the field.

2.1.2. If no decontamination is performed in the field, collect precleaned equipment blanks if the equipment is not certified clean by the vendor or the laboratory providing the equipment.

2.1.3. Equipment blanks are not required for volatile organic compounds.

2.2. Field Blanks:

2.2.1. Collect field blanks if no equipment except the sample container is used to collect the samples or if the sampling equipment is certified clean by the vendor or the laboratory providing the equipment.

2.2.1.1. If a sample container is used as an intermediate sample collection device, collect an equipment blank by rinsing the decontaminated collection container as the substitute for the field blank.

2.2.2. Field blanks are not required for volatile organic compounds.

2.3. Trip Blanks:

2.3.1. These blanks are applicable if samples are to be analyzed for volatile organic compounds. See FQ 1213 for frequency, preparation and handling requirements.

3. OPTIONAL QUALITY CONTROL MEASURES

3.1. The method or project may require collection of additional quality control measures as outlined in FQ 1210 (Blanks), FQ 1220 (Duplicates) and FQ 1240 (Split Samples).

FQ 1240. SPLIT SAMPLES

The DEP or the client may require split samples as a means of determining compliance or as an added measure of quality control. Unlike duplicate samples that measure the variability of both the sample collection and laboratory procedures, split samples measure only the variability **between** laboratories. Therefore, the laboratory samples must be subsamples of the same parent sample and every attempt must be made to ensure sample homogeneity.

Collect, preserve, transport and document split samples using the same protocols as the related samples. In addition, attempt to use the same preservatives (if required).

If split samples are incorporated as an added quality control measure, the DEP recommends that all involved parties agree on the logistics of collecting the samples, the supplier(s) of the preservatives and containers, the analytical method(s), and the statistics that will be used to evaluate the data.

FQ 1241. *Soils, Sediments, Chemical Wastes and Sludges*

Collecting split samples for these matrices is not recommended because a true split sample in these matrices is not possible.

FQ 1242. *Water*

Collect split samples for water in one of two ways:

1. Mix the sample in a large, appropriately precleaned, intermediate vessel (a churn splitter is recommended). This method shall not be used if volatile or extractable organics, oil and grease or total petroleum hydrocarbons are of interest. While continuing to thoroughly mix the sample, pour aliquots of the sample into the appropriate sample containers. Alternatively:

2. Fill the sample containers from consecutive sample volumes **from the same sampling device**. If the sampling device does not hold enough sample to fill the sample containers, use the following procedure:

2.1. Fill the first container with half of the sample, and pour the remaining sample into the second container.

2.2. Obtain an additional sample, pour the first half into the **second** container, and pour the remaining portion into the first container.

2.3. Continue with steps described in sections 2.1 and 2.2 above until both containers are filled.

FQ 1250. QUALITY CONTROL DOCUMENTATION

1. Document all field quality control samples in the permanent field records.
2. At a minimum, record the following information:

- The type, time and date that the quality control sample was collected; and
- The preservative(s) (premeasured or added amount) and preservation checks performed.

3. If blanks are collected/prepared by the field organization, maintain records of the following:
   - Type of analyte-free water used;
   - Source of analyte-free water (include lot number if commercially purchased);
   - A list of the sampling equipment used to prepare the blank.

   If items above are specified in an internal SOP, you may reference the SOP number and revision date in the field notes. Note any deviations to the procedure in the field notes.
4. For trip blanks, record the following:
   - Date and time of preparation
   - Storage conditions prior to release to the sample collecting organization
   - Type of analyte-free water used
   - Source and lot number (if applicable) of analyte-free water

   4.1. Include trip blank information in the sampling kit documentation per FD 2000, section 2.
5. For duplicates, record the technique that was used to collect the sample.
6. For split samples, identify the method used to collect the samples and the source(s) of the sample containers and preservatives.

FS 1000. GENERAL SAMPLING PROCEDURES

See also the following Standard Operating Procedures:

- FA 1000 and 2000 Administrative Procedures
- FC 1000 Cleaning/Decontamination Procedures
- FD 1000-9000 Documentation Procedures
- FM 1000 Field Planning and Mobilization
- FQ 1000 Field Quality Control Requirements

FS 1001. *Preliminary Activities*

1. Begin each sampling trip with some planning and coordination. Refer to FM 1000 for recommendations and suggestions on laboratory selection and communication, and field mobilization.

1.1. DEP recommends that a minimum of two people be assigned to a field team. In addition to safety concerns, the process of collecting the samples, labeling the containers and completing the field records is much easier if more than one person is present.

1.2. If responding to incidents involving hazardous substances, DEP recommends that four or five people be assigned to the team.

2. EQUIPMENT

2.1. Select appropriate equipment based on the sampling source (see FS 2000 to FS 8200), the analytes of interest and the sampling procedure.

2.1.1. If properly cleaned, sample containers may be used as collection devices or intermediate containers.

2.2. The equipment construction must be consistent with the analytes or analyte groups to be collected (see Tables FS 1000-1 and FS 1000-2).

2.3. Bring precleaned equipment to the field or use equipment that has been certified clean by the vendor or laboratory.

3. DEDICATED EQUIPMENT STORAGE

3.1. Store all dedicated equipment (except dedicated pump systems or dedicated drop pipes) in a controlled environment.

3.2. If possible, store equipment in an area that is located away from the sampling site. If equipment other than dedicated pumps or dedicated drop pipes is stored in monitoring wells, suspend the equipment above the formation water.

3.3. Securely seal the monitoring well in order to prevent tampering between sampling events.

3.4. Decontaminate all equipment (except dedicated pumps or drop pipes) before use according to the applicable procedures in FC 1000.

4. SAMPLE CONTAINERS

4.1. The analyses to be performed on the sample determine the construction of sample containers.

4.2. Inspect all containers and lids for flaws (cracks, chips, etc.) before use. Do not use any container with visible defects or discoloration.

FS 1002. *Contamination Prevention and Sample Collection Order*

1. CONTAMINATION PREVENTION

1.1. Take special effort to prevent cross contamination and contamination of the environment when collecting samples. Protect equipment, sample containers and supplies from accidental contamination.

1.1.1. Do not insert pump tubing, measurement probes, other implements, fingers, etc. into sample containers or into samples that have been collected for laboratory analysis.

1.1.1.1. If it is necessary to insert an item into the container or sample, ensure that the item is adequately decontaminated for the analytes of interest to be analyzed in the sample.

1.1.2. If possible, collect samples from the least contaminated sampling location (or background sampling location) to the most contaminated sampling location.

1.1.2.1. Collect the ambient or background samples first and store them in separate ice chests or shipping containers.

1.1.3. Collect samples in flowing water from downstream to upstream.

1.1.4. Do not store or ship highly contaminated samples (concentrated wastes, free product, etc.) or samples suspected of containing high concentrations of contaminants in the same ice chest or shipping container with other environmental samples.

1.1.4.1. Isolate these sample containers by sealing them in separate, untreated plastic bags immediately after collecting, preserving, labeling, etc.

1.1.4.2. Use a clean, untreated plastic bag to line the ice chest or shipping container.

2. SAMPLE COLLECTION ORDER

2.1. Sampling order is a recommendation to be modified depending on site circumstances. Unless field conditions justify other sampling regimens, collect samples in the following order:

- Volatile Organics and Volatile Inorganics
- Extractable Organics, Petroleum Hydrocarbons, Aggregate Organics and Oil & Grease
- Total Metals
- Dissolved Metals
- Inorganic Nonmetallics, Physical and Aggregate Properties, and Biologicals
- Radionuclides
- Microbiological

Note: If the pump used to collect groundwater samples cannot be used to collect volatile or extractable organics, then collect all other parameters, withdraw the pump and tubing, and collect the volatile and extractable organics.

3. COMPOSITE SAMPLES

3.1. Do not collect composite samples unless required by permit or DEP program.

3.2. If compositing is required, use the following procedure:

3.2.1. Select sampling points from which to collect each aliquot.

3.2.2. Using the appropriate sampling technique, collect equal aliquots (same sample size) from each location and place in a properly cleaned container.

3.2.3. Record the approximate amount of each aliquot (volume or weight).

3.2.4. Add preservative(s), if required.

3.2.5. Label container and make appropriate field notes (see FD 1000-9000).

3.2.6. Notify the laboratory that the sample is a composite sample.

3.2.7. When collecting soil or sediment samples, combine the aliquots of the sample directly in the sample container with no pre-mixing. Notify the laboratory that the sample is an unmixed composite sample, and request that the laboratory thoroughly mix the sample before sample preparation or analysis.

3.2.8. When collecting water composites see FS 2000, section 1.3 or pertinent sections of other water matrix SOPs for specific details on collection.

FS 1003. *Protective Gloves*

1. Gloves serve a dual purpose to:

- Protect the sample collector from potential exposure to sample constituents
- Minimize accidental contamination of samples by the collector

2. The DEP recommends wearing protective gloves when conducting all sampling activities. They must be worn except when:

- The sample source is considered to be non-hazardous
- The samples will not be analyzed for trace constituents
- The part of the sampling equipment that is handled without gloves does not contact the sample source

3. Do not let gloves come into contact with the sample or with the interior or lip of the sample container.

4. Use clean, new, unpowdered and disposable gloves.

4.1. DEP recommends latex gloves, however, other types of gloves may be used as long as the construction materials do not contaminate the sample or if internal safety protocols require greater protection.

4.2. Note that certain materials (as might be potentially present in concentrated effluent) may pass through certain glove types and be absorbed in the skin. Many vendor catalogs provide information about the permeability of different gloves and the circumstances under which the glove material might be applicable.

4.3. The powder in powdered gloves can contribute significant contamination and DEP does not recommend wearing powdered gloves unless it can be demonstrated that the powder does not interfere with the sample analysis.

5. If gloves are used, change:
    - After preliminary activities such as pump placement;
    - After collecting all the samples at a single sampling point; or
    - If torn, or used to handle extremely dirty or highly contaminated surfaces.
6. Properly dispose of all used gloves.

FS 1004. *Container and Equipment Rinsing*

When collecting aqueous samples, rinse the sample collection equipment with a portion of the sample water before taking the actual sample. Sample containers do not need to be rinsed. In the case of petroleum hydrocarbons, oil & grease or containers with premeasured preservatives, the sample containers cannot be rinsed.

FS 1005. *Fuel-Powered Equipment and Related Activities*

1. Place all fuel-powered equipment away from, and downwind of, any site activities (e.g., purging, sampling, decontamination). If field conditions preclude such placement (i.e., the wind is from the upstream direction in a boat), place the fuel source(s) as far away as possible from the sampling activities and describe the conditions in the field notes.

2. Handle fuel (i.e., filling vehicles and equipment) prior to the sampling day. If such activities must be performed during sampling, the personnel must wear disposable gloves. Dispense all fuels, dispose of gloves downwind, and well away from the sampling activities.

3. If sampling at active gas stations, stop sample collection activities during fuel deliveries.

FS 1006. *Preservation, Holding Times and Container Types*

1. Preserve all samples according to the requirements specified in Tables FS 1000-4 through FS 1000-10.

    1.1. The information listed in the above-referenced tables supersedes any preservation techniques, holding time or container type that might be discussed in individual analytical methods.

    1.2. If samples are collected only for total phosphorus and are not for NPDES compliance, thermal preservation (ice) is not required if the sample containers are pre-preserved with acid.

2. The preservation procedures in the referenced tables specify immediate preservation. "Immediate" is defined as "within 15 minutes of sample collection." Perform all preservation on-site (in the field).

    2.1. Preservation is not required if samples can be transported back to the laboratory within 15 minutes of collecting the sample and

        2.1.1. The laboratory begins sample analysis within the 15-minute window and documents the exact time the analysis began, or

        2.1.2. The laboratory adds the appropriate preservatives (including thermal preservation) within 15 minutes of sample collection and documents the exact time that the preservation was done.

3. PRESERVING COMPOSITE WATER SAMPLES

3.1. If the sample preservation requires thermal preservation (e.g., <6°C), the samples must be cooled to the specified temperature.

3.1.1. Manually collected samples to be composited must be refrigerated at a temperature equal to or less than the required temperature.

3.1.2. Automatic samplers must be able to maintain the required temperature by packed ice or refrigeration.

3.2. When chemical preservation is also required, begin the preservation process within 15 minutes of the last collected sample.

3.3. Holding Times for Automatic Samplers:

3.3.1. If the collection period is 24 hours or less, the holding time begins at the last scheduled sample collection;

3.3.2. If the collection period exceeds 24 hours, the holding time begins with the time that the first sample is collected.

4. pH ADJUSTED PRESERVATION - Check the pH of pH-adjusted samples according to these frequencies:

4.1. During the first sampling event at a particular site, check **all** samples (includes each groundwater monitoring well, surface water location, or influent/effluent sampling location) that are pH-adjusted except volatile organics.

4.2. During subsequent visits to a particular site, check at least one sample per parameter group that must be pH-adjusted.

4.3. If the frequency of sample collection at a specified location is greater than once per month (i.e., weekly or daily), check the pH of at least one sample per parameter group (except volatile organics) according to the following schedule:

4.3.1. Weekly sampling: 1 pH check per month

4.3.2. Daily sampling: 1 pH check per week

4.4. If the frequency of sample collection at a specified location is once per month, check the pH of at least one sample per parameter group (except volatile organics) quarterly.

4.5. If site conditions vary from sampling event to sampling event, perform pH checks at increased intervals.

5. THERMAL PRESERVATION

5.1. When preservation requirements indicate cooling to a specific temperature, samples must be placed in wet ice within 15 minutes of sample collection (see 1006, section 2 above). Unless specified, do not freeze samples.

5.2. All supplies (ice, dry ice, etc.) necessary to meet a thermal preservation requirement must be onsite for immediate use.

5.3. Ship samples in wet ice. If samples are cooled to the required temperature before shipment, samples may be shipped with frozen ice packs if the specified temperature is maintained during shipment. The sample temperature must not exceed the specified temperature.

5.4. If immediate freezing is required, dry ice must be available in the field to begin the freezing process.

FS 1007. *Preventive and Routine Maintenance*

Preventive maintenance activities are necessary to ensure that the equipment can be used to obtain the expected results and to avoid unusable or broken equipment while in the field. Equipment is properly maintained when:

- It functions as expected during mobilization; and
- It is not a source of sample contamination (e.g., dust).

1. Follow the manufacturer's suggested maintenance activities and document all maintenance. At a minimum, DEP recommends the activities listed on Table FS 1000-12.

2. Maintain documentation for the following information for each piece of equipment or instrumentation. See FD 3000 also.

2.1. Designate the identity of specific instrumentation in the documentation with a unique description or code for each instrument unit employed. This identifier may include a manufacturer name, model number, serial number, inventory number or other unique identification.

2.2. Log all maintenance and repair performed for each instrument unit, including routine cleaning procedures and solution or parts replacement for instrument probes.

2.3. Include the calendar date for the procedures performed.

2.4. Record names of personnel performing the maintenance or repair tasks.

2.5. Describe any malfunctions necessitating repair or service.

2.6. Retain vendor service records for all affected instruments.

2.7. Record the following for rented equipment:

- Rental date(s)
- Equipment type and model or inventory number or other description

2.8. Retain the manufacturer’s operating and maintenance instructions.

FS 1008. *Documentation and References*

1. REFERENCES: All sampling references must be available for consultation in the field. These include:

- DEP SOPs;
- Internal SOPs;
- Sampling and analysis plans; and/or
- Quality Assurance Project Plans.

2. DOCUMENTATION: Complete and sign all documentation (see FD 1000).

FS 1009. *Sample Documentation and Evidentiary Custody*

1. SAMPLE DOCUMENTATION

1.1. Document all activities related to a sampling event, including sample collection, equipment calibration, equipment cleaning and sample transport.

1.2. The required documentation related to each sampling or other field activity is specified in the associated SOPs; i.e., FQ 1000, FC 1000, the FS series, and the FT series.

1.3. The documentation requirements are also summarized in FD 1000, Field Documentation. FD 1000 additionally contains a list of example forms published with the SOPs that may be used to document various activities or as templates for creating customized forms.

2. LEGAL CHAIN OF CUSTODY (COC)

The use of legal or evidentiary Chain-of-Custody (COC) protocols is not usually required by DEP, except for cases involving civil or criminal enforcement. Do not use these procedures for routine sampling for compliance unless evidentiary custody protocols are specifically mandated in a permit or other legal order or when required for enforcement actions.

Evidentiary sample custody protocols are used to demonstrate that the samples and/or sample containers were handled and transferred in such a manner as to eliminate possible tampering.

When a client or situation requires legal COC, use the procedures in FD 7000 to document and track all time periods associated with the physical possession and storage of sample containers, samples, and subsamples from point of origin through the final analytical result and sample disposal.

When legal or evidentiary COC is required, samples must be:

- In the actual possession of a person who is authorized to handle the samples (e.g., sample collector, laboratory technician);
- In the view of the same person after being in their physical possession;
- Secured by the same person to prevent tampering; or
- Stored in a designated secure area.

2.1. Control and document access to all evidentiary samples and subsamples with adequate tracking. Documentation must include records about each of the activities and situations listed below, when applicable to sample evidence, and must track the location and physical handling of all samples by all persons at all times.

2.1.1. Limit the number of individuals who physically handle the samples as much as practicable.

2.1.2. When storing samples and subsamples, place samples in locked storage (e.g., locked vehicle, locked storeroom, etc.) at all times when not in the possession or view of authorized personnel.

2.1.3. Alternatively, maintain restricted access to facilities where samples are stored. Ensure that unauthorized personnel are not able to gain access to the samples at any time.

2.1.4. Do not leave samples in unoccupied motel or hotel rooms or other areas where access cannot be controlled by the person(s) responsible for custody without first securing samples and shipping or storage containers with tamper-indicating evidence tape or custody seals. Ice chests or other storage containers used to store sample containers in hotel rooms may be sealed instead of sealing each sample container stored within.

2.2. Use a Chain of Custody form or other transmittal record to document sample transfers to other parties. Other records and forms may be used to document internal activities if they meet the requirements for legal chain of custody.

2.3. Legal COC begins when the precleaned sample containers are dispatched to the field.

2.3.1. The person who relinquishes the prepared sample kits or containers and the individual who receives the sample kits or containers must sign the COC form unless the same party provides the containers and collects the samples.

2.3.2. All parties handling the empty sample containers and samples are responsible for documenting sample custody, including relinquishing and receiving samples, except commercial common carriers.

2.4. Shipping Samples under Legal COC

2.4.1. Complete all relevant information on the COC transmittal form or record (see FD 7200, section 2).

2.4.2. Internal records must document the handling of the samples and shipping containers in preparation for shipment. The names of all persons who have prepared the shipment must be recorded. All time intervals associated with handling and preparation must be accounted for.

2.4.3. Place the forms in a sealed waterproof bag and place in the shipping container with the samples.

2.4.4. Seal the shipping container with tamper-proof seals (see 2.6 below) so that any tampering can be clearly seen by the individual who receives the samples.

2.4.5. Note: The common carrier does not sign COC records. However, the common carrier (when used) must be identified.

2.5. Delivering Samples to the Laboratory

2.5.1. All individuals who handle and relinquish the sample containers must sign the transmittal form. The legal custody responsibilities of the field operations end when the samples are relinquished to the laboratory.

2.6. Chain of Custody Seals: If required, affix tamper-indicating evidence tape or seals to all sample, storage and shipping container closures when transferring or shipping sample container kits or samples to another party.

2.6.1. Place the seal so that the closure cannot be opened without breaking the seal.

2.6.2. Record the time, calendar date and signatures of responsible personnel affixing and breaking all seals for each sample container and shipping container.

2.6.3. Affix new seals every time a seal is broken until continuation of evidentiary custody is no longer required.

FS 1010. *Health and Safety*

Implement all local, state and federal requirements relating the health and safety.

FS 1011. *Hazardous Wastes*

Follow all local, state and federal requirements pertaining to the storage and disposal of any hazardous or investigation-derived wastes.

1. Properly manage all investigation-derived waste (IDW) so contamination is not spread into previously uncontaminated areas.

1.1. IDW includes all water, soil, drilling mud, decontamination wastes, discarded personal protective equipment (PPE), etc. from site investigations, exploratory borings, piezometer and monitoring well installation, refurbishment, and abandonment, and other investigative activities. Containerize the IDW at the time it is generated.

1.2. Determine if the IDW must be managed as Resource Conservation and Recovery Act (RCRA) regulated hazardous waste through appropriate testing or generator knowledge. Manage all IDW that is determined to be RCRA regulated hazardous waste according to the local state and federal requirements.

1.3. Properly dispose of IDW that is not a RCRA-regulated hazardous waste but is contaminated above the Department's Soil Cleanup Target Levels or the state standards and/or minimum criteria for ground water quality.

1.4. IDW that is not contaminated or contains contaminants below the Department's Soil Cleanup Target Levels or the state standards and/or minimum criteria for ground water quality may be disposed of onsite as long as the IDW will not cause a surface water violation.

1.5. Maintain all containers holding IDW in good condition:

1.5.1. Periodically inspect the containers for damage

1.5.2. Ensure that all required labeling (DOT, RCRA, etc.) are clearly visible.

Appendix FS 1000
Tables, Figures and Forms

Table FS 1000-1

Equipment Construction Materials

Construction Material[1]	Acceptable Analyte Groups	Precautions
Metals		
316 Stainless Steel	All analyte groups. Recommended for inorganic nonmetallics, metals, volatile and extractable organics.	Do not use if weathered, corroded or pitted.[2]
300-Series Stainless Steel (304, 303, 302)	Suitable for all analyte groups (if used, check for corrosion before use). Recommended for inorganic nonmetallics, metals, volatile and extractable organics.	Do not use if weathered, corroded or pitted.[2] If corroded, there is a potential for samples to be contaminated with iron, chromium, copper or nickel. Check for compatibility with water chemistry for dedicated applications. Do not use in low pH, high chloride, or high TDS waters.
Low Carbon Steel Galvanized Steel Carbon Steel	Inorganic nonmetallics only.	Coring devices are acceptable for all analyte groups **if** appropriate liners are used. Use Teflon liners for organics. Use plastic or Teflon liners for metals. Do not use if weathered, corroded or pitted.[2] If corroded, there is a potential for samples to be contaminated with iron and manganese. Galvanized equipment will also contaminate with zinc and cadmium. If used to collect large samples (e.g., dredges), collect organic and metal samples may be collected from portions of the interior of the collected material.
Brass	Inorganic nonmetallics only.	Do not use if weathered, corroded or pitted.[2]
Plastics[3]		
Teflon and other fluorocarbon polymers	All analyte groups. Especially recommended for trace metals and organics.	Easily scratched. Do not use if scratched or discolored.
Polypropylene Polyethylene (All Types)	All analyte groups.	Easily scratched. Do not use if scratched or discolored.
Polyvinyl chloride (PVC)	All analyte groups except extractable and volatile organics.	Do not use when collecting extractable or volatile organics samples.

Table FS 1000-1

Equipment Construction Materials

Construction Material[1]	Acceptable Analyte Groups	Precautions
Tygon, Silicone, Neoprene	All analyte groups except extractable and volatile organics.	Do not use when collecting extractable or volatile organic samples. Do not use silicone if sampling for silica.
Viton	All analyte groups except extractable and volatile organics.[4]	Minimize contact with sample. Use only if no alternative material exists.
Glass		
Glass, borosilicate	All analyte groups except silica and boron.	

Adapted from USGS Field Manual, Chapter 2, January 2000.

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[1] Refers to construction material of the portions of the sampling equipment that come in contact with the sample (e.g., housing of variable speed submersible pump must be stainless steel if extractable organics are sampled; the housing of a variable speed submersible pump used to sample metals may be plastic.)

[2] Corroded/weathered surfaces are active sorption sites for organic compounds.

[3] Plastics used in connection with inorganic trace element samples (including metals) must be uncolored or white.

[4] May be allowable for specialized parts where no alternative material exists (e.g., Viton seals are the best available seal for some dedicated pump systems), however, contact with the sample must be minimized.

Table FS 1000-2
Construction Material Selection for Equipment and Sample Containers

Analyte Group	Acceptable Materials
Extractable Organics	Teflon Stainless steel Glass Polypropylene (All types) Polyethylene (All types) All parts of the system including connectors and gaskets must be considered – Viton may be used if no other material is acceptable.
Volatile Organics	Teflon Stainless steel Glass Polypropylene (All types) Polyethylene (All types) All parts of the system including connectors and gaskets must be considered – Viton may be used if no other material is acceptable.
Metals	Teflon Stainless steel Polyethylene (All types) Polypropylene (All types) Tygon, Viton, Silicone, Neoprene PVC Glass (except silica and boron)
Ultratrace Metals	Teflon Polyethylene (All types) Polypropylene (All types) Polycarbonate Mercury must be in glass or Teflon
Inorganic Nonmetallics	Teflon Stainless steel Low carbon, Galvanized or Carbon steel Polyethylene (All types) Polypropylene (All types) Tygon, Viton, Silicone, Neoprene PVC Glass Brass

Table FS 1000-2
Construction Material Selection for Equipment and Sample Containers

Analyte Group	Acceptable Materials
Microbiological samples	Teflon Stainless steel Polyethylene (All types) Polypropylene (All types) Tygon, Viton, Silicone, Neoprene PVC Glass Sterilize all **sample** containers. Thoroughly clean **sampling equipment** and rinse several times with sample water before collection. Sampling equipment **does not require** sterilization **Do not rinse sample containers**

Table FS 1000-3
Equipment Use and Construction

EQUIPMENT	CONSTRUCTION HOUSING[1]	CONSTRUCTION TUBING	USE	PERMISSIBLE ANALYTE GROUPS	RESTRICTIONS AND PRECAUTIONS
WATER SAMPLING					
GROUNDWATER					
1 Positive displacement pumps[2]					
a. Submersible (turbine, helical rotor, gear driven)	SS, Teflon	SS, Teflon, PE, PP	Purging	All analyte groups	[3,4,5]; must be variable speed
			Sampling	All analyte groups	[3,4,5] must be variable speed
	SS, Teflon	Non-inert[6]	Purging	All analyte groups	[3,4,5] must be variable speed; polishing required[7]
			Sampling	All analyte groups except volatile and extractable organics	Must be variable speed If sampling for metals, the tubing must be non-metallic if not SS
	Non-inert[6]	Non-inert[6]	Purging	All analyte groups	[3,4,5] must be variable speed; polishing required[7]
			Sampling	All analyte groups except volatile and extractable organics	Must be variable speed If sampling for metals, the tubing must be non-metallic if not SS
b. Bladder pump (no gas contact)	SS, Teflon, PE, PP or PVC if permanently installed	SS, Teflon, PE, PP	Purging	All analyte groups	[3,4,5] must be variable speed
			Sampling	All analyte groups	[3,4] must be variable speed Bladder must be Teflon if sampling for volatile or extractable organics or PE or PP if used in portable pumps
	SS, Teflon, PE, PP	Non-inert[6]	Purging	All analyte groups	[3,4] must be variable speed; polishing required[7] **This configuration is not recommended**
			Sampling	All analyte groups except volatile and extractable organics	[3,4] must be variable speed If sampling for metals, the tubing must be non-metallic if not SS
	Non-inert[6]	Non-inert[6]	Purging	All analyte groups	[3,4] must be variable speed; polishing required[7]
			Sampling	All analyte groups except volatile and extractable organics	[3,4] must be variable speed; polishing required[7] If sampling for metals, the tubing must be non-metallic if not SS

Table FS 1000-3
Equipment Use and Construction

EQUIPMENT	CONSTRUCTION HOUSING[1]	CONSTRUCTION TUBING	USE	PERMISSIBLE ANALYTE GROUPS	RESTRICTIONS AND PRECAUTIONS
2. Suction lift pumps					
a. Centrifugal	N/A	SS, Teflon, PE, PP	Purging	All analyte groups	[4] foot-valve required Must be variable speed
	N/A	Non-inert[6]	Purging	All analyte groups	[4] foot-valve required; polishing required[7] Must be variable speed
b. Peristaltic	N/A	SS, Teflon, PE, PP	Purging	All analyte groups	[4] foot-valve required; polishing required[7] or continuous pumping required Must be variable speed
			Sampling	All analyte groups except volatile organics	[4] Silicone tubing in pump head Must be variable speed
	N/A	Non-inert[6]	Purging	All analyte groups	[4] foot-valve required Must be variable speed
			Sampling	All analyte groups except volatile and extractable organics	[4] Silicone tubing in pump head Must be variable speed
3. Bailers	SS, Teflon, PE, PP	N/A	Purging	All analyte groups	None; **not recommended**
		N/A	Sampling	All analyte groups	None; **not recommended**
	Non-inert[6]	N/A	Purging	All analyte groups except volatile and extractable organics	None; **not recommended** If sampling for metals, the tubing must be non-metallic if not SS
			Sampling	All analyte groups except volatile and extractable organics	None; **not recommended** If sampling for metals, the tubing must be non-metallic if not SS
SURFACE WATER					
1. Intermediate containers such as pond sampler, scoops, beakers, buckets, and dippers	SS, Teflon, Teflon-coated, PE, PP	N/A	Grab sampling	All analyte groups	None
	Glass	N/A		All analyte groups except boron and fluoride	None
	Non-inert[6]	N/A		All analyte groups except volatile and extractable organics	None

Table FS 1000-3
Equipment Use and Construction

EQUIPMENT	CONSTRUCTION		USE	PERMISSIBLE ANALYTE GROUPS	RESTRICTIONS AND PRECAUTIONS
	HOUSING[1]	TUBING			
2. Nansen, Kemmerer, Van Dorn, Alpha and Beta Samplers, Niskin (or equivalent)	SS, Teflon, Teflon-coated, PE, PP	N/A	Specific depth grab sampling	All analyte groups	None
	Non-inert[6]	N/A		All analyte groups except volatile and extractable organics	None
3. DO Dunker	SS, Teflon, glass, PE, PP	N/A	Water column composite sampling	All analyte groups	None
4 Bailers – double valve	SS, Teflon, PE, PP	N/A	Grab sampling	All analyte groups	None
	Non-inert[6]	N/A	Grab sampling	All analyte groups except volatile and extractable organics	None If sampling for metals, the tubing must be non-metallic if not SS
5. Peristaltic pump	N/A	SS, Teflon, PE, PP	Specific depth sampling	All analyte groups except volatile organics	Silicone tubing in pump head Must be variable speed
	N/A	Non-inert[6]		All analyte groups except volatile and extractable organics	Silicone tubing in pump head Must be variable speed
FIELD FILTRATION UNITS	N/A		Dissolved constituents	Inorganic nonmetallics and metals in surface water	Must use a 0.45 μm filter
				Inorganic nonmetallics in groundwater	Must use a 0.45 μm filter
				Metals in groundwater and static wastewater and surface water	Must use in-line, high capacity, one-piece molded filter that is connected to the outlet of a pump; no intermediate vessels; positive pressure PE, PP & Teflon bailers acceptable Must use a 1 μm filter in groundwater, a 0.45 μm filter in surface water
				Metals in moving surface water (i.e., river/stream)	Must use positive pressure device, but an intermediate vessel may be used. Use a 0.45 μm filter

Table FS 1000-3
Equipment Use and Construction

EQUIPMENT	CONSTRUCTION HOUSING[1]	CONSTRUCTION TUBING	USE	PERMISSIBLE ANALYTE GROUPS	RESTRICTIONS AND PRECAUTIONS
SOLID SAMPLING					
SOILS					
1. Core barrel (or liner)	SS, Teflon, glass, Teflon-coated, aluminum, PE, PP	N/A	Sampling	All analyte groups[8]	[9, 10, 11]
	Non-inert[6] nonmetallics	N/A	Sampling	All analyte groups	[12]
	Non-inert[6] metals	N/A	Sampling	All analyte groups	[12]
2. Trowel, scoop, spoon or spatula	SS, Teflon, Teflon-coated, PE, PP	N/A	Sampling	All analyte groups[8]	
			Compositing	All analyte groups except volatile organics	Samples for volatile organics must grab samples
	Plastic	N/A	Sampling and compositing	All analyte groups except volatile and extractable organics	None Must be nonmetallic if not SS
3. Mixing tray (pan)	SS, Teflon, glass, Teflon-coated, aluminum , PE, PP	N/A	Sampling	All analyte groups[8]	[11]
			Compositing or homogenizing	All analyte groups except volatile organics	[11]
	Non-inert[6]	N/A	Compositing or homogenizing	All analyte groups	[10,11,12] must be nonmetallic if not SS
4. Shovel, bucket auger	SS	N/A	Sampling	All analyte groups[8]	None
	Non-SS	N/A	Sampling	All analyte groups[8]	[10,11,12]
5. Split spoon	SS or carbon steel w/ Teflon insert	N/A	Sampling	All analyte groups[8]	[10,11,12]
6. Shelby tube	SS	N/A	Sampling	All analyte groups[8]	[9]
	Carbon steel	N/A	Sampling	All analyte groups	[9,10,12]
SEDIMENT					
1. Coring devices	SS, Teflon, glass, Teflon-coated, aluminum, PE, PP	N/A	Sampling	All analyte groups[8]	[9,10,11]

Table FS 1000-3
Equipment Use and Construction

EQUIPMENT	CONSTRUCTION HOUSING[1]	CONSTRUCTION TUBING	USE	PERMISSIBLE ANALYTE GROUPS	RESTRICTIONS AND PRECAUTIONS
	Non-inert[6] nonmetallics	N/A	Sampling	All analyte groups	[12]
	Non-inert[6] metals	N/A	Sampling	All analyte groups	[9,10,11]
2. Grab – Young, Petersen, Shipek	Teflon, Teflon-lined, SS	N/A	Sampling	All analyte groups[8]	None
	Carbon steel	N/A	Sampling	All analyte groups	[10,11]
3. Dredges – Eckman, Ponar, Petit Ponar Van Veen	SS	N/A	Sampling	All analyte groups[8]	None
	Carbon steel, brass	N/A	Sampling	All analyte groups	[10,11]
4. Trowel, scoop, spoon or spatula	SS, Teflon, Teflon-coated, PE, PP	N/A	Sampling	All analyte groups[8]	
			Compositing	All analyte groups except volatile organics	Samples for volatile organics be grab samples
	Plastic	N/A	Sampling and compositing	All analyte groups except volatile and extractable organics	None must be nonmetallic if not SS
5. Mixing tray (pan)	SS, Teflon, glass, Teflon-coated, aluminum, PE, PP	N/A	Sampling	All analyte groups[8]	[11]
			Compositing or homogenizing	All analyte groups except volatile organics	[11]
	Non-inert[6]	N/A	Compositing or homogenizing	All analyte groups except volatile and extractable organics	none [11] must be nonmetallic if not SS
WASTE[13]					
Scoop	SS	N/A	Liquids, solids & sludges	All analyte groups[8]	Cannot collect deeper phases
Spoon	SS	N/A	Solids, sludges	All analyte groups[8]	Cannot collect deeper phases
Push tube	SS	N/A	Solids, sludges	All analyte groups[8]	Cannot collect deeper phases
Auger	SS	N/A	Solids	All analyte groups[8]	None

Table FS 1000-3
Equipment Use and Construction

EQUIPMENT	CONSTRUCTION		USE	PERMISSIBLE ANALYTE GROUPS	RESTRICTIONS AND PRECAUTIONS
	HOUSING[1]	TUBING			
Sediment sampler	SS	N/A	Impoundments, piles	All analyte groups[8]	None
Ponar dredge	SS	N/A	Solids, sludges & sediments	All analyte groups[8]	None
Coliwasa, Drum thief	Glass	N/A	Liquids, sludges	All analyte groups	None
Mucksucker, Dipstick	Teflon		Liquids, sludges	All analyte groups	Not recommended for tanks > 11 feet deep
Bacon bomb	SS	N/A	Liquids	All analyte groups[8]	Not recommended for viscous wastes
Bailer	SS, Teflon	N/A	Liquids	All analyte groups[8]	Do not use with heterogeneous wastes Not recommended for viscous wastes
Peristaltic pump	N/A	Teflon, Glass	Liquids	All analyte groups except volatile organics	Do not use in flammable atmosphere Not recommended for viscous wastes
Backhoe bucket	Steel	N/A	Solids, Sludges		Difficult to clean Volatiles and metals must be taken from the interior part of the sample
Split spoon	SS	N/A	Solids	All analyte groups[8]	
Roto-Hammer	Steel	N/A	Solids	All analyte groups[8]	Physically breaks up sample Not for flammable atmospheres

Acronyms:

N/A	not applicable
SS	stainless steel
HDPE	high-density polyethylene
PE	polyethylene
PVC	polyvinyl chloride
PP	polypropylene

Table FS 1000-3
Equipment Use and Construction

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[1] Refers to tubing and pump housings/internal parts that are in contact with purged or sampled water (interior and exterior of delivery tube, inner lining of the discharge tube, etc.).

[2] If used to collect volatile or extractable organics, all power cords and other tubing must be encased in Teflon, PE or PP.

[3] If used as a non-dedicated system, pump must be completely disassembled, if practical, and cleaned between wells.

[4] Delivery tubing must be precleaned and precut at the base of operations or laboratory. If the same tubing is used during the sampling event, it must be cleaned and decontaminated between uses.

[5] In-line check valve required.

[6] "Non-inert" pertains to materials that are reactive (adsorb, absorb, etc.) to the analytes being sampled. For organics, materials include rubber, plastics (except PE and PP), and PVC. For metals, materials include brass, galvanized, and carbon steel.

[7] "Polishing": When purging for volatile or extractable organics, the entire length of tubing or the portion which comes in contact with the formation water must be constructed of Teflon, SS, PE or PP. If other materials (e.g., PVC, garden hoses, etc.) are used, the following protocols must be followed: 1) slowly withdraw the pump from the water column during the last phase of purging, to remove any water from the well that may have contacted the exterior of the pump and/or tubing; 2) remove a single well volume with the sampling device before sampling begins. **Do not use Tygon** for purging if purgeable or extractable organics are of interest. Polishing **is not recommended;** use of sampling equipment constructed of appropriate materials is preferred.

[8] Do not use if collecting for hexavalent chromium ($Chromium^{+6}$)

[9] If samples are sealed in the liner for transport to the laboratory, the sample for VOC analysis must be taken from the interior part of the core.

[10] If a non-stainless steel (carbon steel, aluminum) liner, core barrel or implement is used, take the samples for metals, purgeable organics and organics from the interior part of the core sample.

[11] Aluminum foil, trays or liners may be used only if aluminum is not an analyte of interest.

[12] If non-inert-liner, core barrel or implement is used, take samples from the interior part of the collected sample.

[13] If disposable equipment of alternative construction materials is used, the construction material must be compatible with the chemical composition of the waste, cannot alter the characteristics of the waste sample in any way, and cannot contribute analytes of interest or any interfering components.

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Applicable to **all** Non-Potable Water Samples (includes wastewater, surface water, and groundwater)

Parameter No./Name (refers to parameter number on Tables IA,B, C, D,E, F, G & H as noted)	Container[1]	Preservation[2, 3]	Maximum holding time[4]
Table IA—Bacterial Tests:			
1–5. Coliform, total, fecal, and *E. coli*	PA, G	Cool, <10 °C, 0.0008% $Na_2S_2O_3$ [5]	6 hours[6, 7]
6. Fecal streptococci	PA, G	Cool, <10 °C, 0.0008% $Na_2S_2O_3$ [5]	6 hours[6]
7. Enterococci	PA, G	Cool, <10 °C, 0.0008% $Na_2S_2O_3$ [5]	6 hours[6]
8. Salmonella	PA, G	Cool, <10 °C, 0.0008% $Na_2S_2O_3$ [5]	6 hours[6]
Table IA— Aquatic Toxicity Tests:			
9–11. Toxicity, acute and chronic	P, FP, G	Cool, ≤6 °C [8]	36 hours
Table IB—Inorganic Tests:			
1. Acidity	P, FP, G	Cool, ≤6 °C[9]	14 days
2. Alkalinity	P, FP, G	Cool, ≤6 °C[9]	14 days
4. Ammonia	P, FP, G	Cool, ≤6 °C[9], H_2SO_4 to pH<2	28 days
9. Biochemical oxygen demand	P, FP, G	Cool, ≤6 °C[9]	48 hours
10. Boron	P, FP, or Quartz	HNO_3 to pH<2	6 months
11. Bromide	P, FP, G	None required	28 days
14. Biochemical oxygen demand, carbonaceous	P, FP G	Cool, ≤6 °C[9]	48 hours
15. Chemical oxygen demand	P, FP, G	Cool, ≤6 °C[9], H_2SO_4 to pH<2	28 days
16. Chloride	P, FP, G	None required	28 days
17. Chlorine, total residual	P, G	None required	Analyze within 15 minutes
21. Color	P, FP, G	Cool, ≤6 °C[9]	48 hours
23–24. Cyanide, total or available (or CATC)	P, FP, G	Cool, ≤6 °C[9], NaOH to pH>12[10], reducing agent [5]	14 days
25. Fluoride	P	None required	28 days
27. Hardness	P, FP, G	HNO_3 or H_2SO_4 to pH<2	6 months
28. Hydrogen ion (pH)	P, FP, G	None required	Analyze within 15 minutes
31, 43. Kjeldahl and organic N	P, FP, G	Cool, ≤6 °C[9], H_2SO_4 to pH<2	28 days
Table IB—Metals:			
7 18. Chromium VI	P, FP, G	Cool, ≤6 °C[9], pH = 9.3–9.7[12]	28 days
35. Mercury (CVAA)	P, FP, G	HNO_3 to pH<2	28 days

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Applicable to **all** Non-Potable Water Samples (includes wastewater, surface water, and groundwater)

Parameter No./Name (refers to parameter number on Tables IA,B, C, D,E, F, G & H as noted)	Container[1]	Preservation[2, 3]	Maximum holding time[4]
35. Mercury (CVAFS)	FP, G; and FP-lined cap[13]	5 mL/L 12N HCl or 5 mL/L BrCl[13]	90 days[13]
3, 5–8, 12, 13, 19, 20, 22, 26, 29, 30, 32–34, 36, 37, 45, 47, 51, 52, 58–60, 62, 63, 70– 72, 74, 75. Metals, except boron, chromium VI, and mercury.	P, FP, G	HNO_3 to pH<2, or at least 24 hours prior to analysis[14]	6 months
38. Nitrate	P, FP, G	Cool, ≤6 °C[9]	48 hours
39. Nitrate-nitrite	P, FP, G	Cool, ≤6 °C[9], H_2SO_4 to pH<2	28 days
40. Nitrite	P, FP, G	Cool, ≤6 °C[9]	48 hours
41. Oil and grease	G	Cool, ≤6 °C[9], HCl or H2SO4 to pH<2	28 days
42. Organic Carbon	P, FP, G	Cool, ≤6 °C[9], HCl, H_2SO_4, or H_3PO_4 to pH<2.	28 days
44. Orthophosphate	P, FP, G	Cool, ≤6 °C[9]	Filter within 15 minutes; Analyze within 48 hours
46. Oxygen, Dissolved Probe	G, Bottle and top	None required	Analyze within 15 minutes
47. Winkler	G, Bottle and top	Fix on site and store in dark	8 hours
48. Phenols	G	Cool, ≤6 °C[9], H_2SO_4 to pH<2	28 days
49. Phosphorous (elemental)	G	Cool, ≤6 °C[9]	48 hours
50. Phosphorous, total	P, FP, G	Cool, ≤6 °C[9], H_2SO_4 to pH<2	28 days
53. Residue, total	P, FP, G	Cool, ≤6 °C[9]	7 days
54. Residue, Filterable	P, FP, G	Cool, ≤6 °C[9]	7 days
55. Residue, Nonfilterable (TSS)	P, FP, G	Cool, ≤6 °C[9]	7 days
56. Residue, Settleable	P, FP, G	Cool, ≤6 °C[9]	48 hours
57. Residue, Volatile	P, FP, G	Cool, ≤6 °C[9]	7 days
61. Silica	P or Quartz	Cool, ≤6 °C[9]	28 days
64. Specific conductance	P, FP, G	Cool, ≤6 °C[9]	28 days
65. Sulfate	P, FP, G	Cool, ≤6 °C[9]	28 days
66. Sulfide	P, FP, G	Cool, ≤6 °C[9], add zinc acetate plus sodium hydroxide to pH>9	7 days
67. Sulfite	P, FP, G	None required	Analyze within 15 minutes
68. Surfactants	P, FP, G	Cool, ≤6 °C[9]	48 hours

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Applicable to **all** Non-Potable Water Samples (includes wastewater, surface water, and groundwater)

Parameter No./Name (refers to parameter number on Tables IA,B, C, D,E, F, G & H as noted)	Container[1]	Preservation[2, 3]	Maximum holding time[4]
69. Temperature	P, FP, G	None required	Analyze
73. Turbidity	P, FP, G	Cool, ≤6 °C[9]	48 hours
Table IC—Organic Tests 8			
13, 18–20, 22, 24–28, 34–37, 39–43, 45–47, 56, 76, 104, 105, 108–111, 113. Purgeable Halocarbons	G, FP-lined septum	Cool, ≤6 °C[9], 0.008% $Na_2S_2O_3$ [5]	14 days
6, 57, 106. Purgeable aromatic hydrocarbons	G, FP-lined septum	Cool, ≤6 °C[9], 0.008% $Na_2S_2O_3$ [5], HCl to pH 2[16]	14 days[16]
3, 4. Acrolein and acrylonitrile	G, FP-lined septum	Cool, ≤6 °C[9], 0.008% $Na_2S_2O_3$ [5], pH to 4–5[17]	14 days[17]
23, 30, 44, 49, 53, 77, 80, 81, 98, 100, 112. Phenols[18]	G, FP-lined cap	Cool, ≤6 °C[9], 0.008% $Na_2S_2O_3$ [5]	7 days until extraction, 40 days after extraction
7, 38. Benzidines [18, 19]	G, FP-lined cap	Cool, ≤6 °C[9], 0.008% $Na_2S_2O_3$ [5]	7 days until extraction[20]
14, 17, 48, 50–52. Phthalate esters [18]	G, FP-lined cap	Cool, ≤6 °C[9]	7 days until extraction, 40 days after extraction
82–84. Nitrosamines [18, 21]	G, FP-lined cap	Cool, ≤6 °C[9], store in dark, 0.008% $Na_2S_2O_3$ [5]	7 days until extraction, 40 days after extraction
88–94. PCBs[18]	G, FP-lined cap	Cool, ≤6 °C[9]	1 year until extraction, 1 year after extraction
54, 55, 75, 79. Nitroaromatics and isophorone[18]	G, FP-lined cap	Cool, ≤6 °C[9], store in dark, 0.008% $Na_2S_2O_3$ [5]	7 days until extraction, 40 days after extraction
1, 2, 5, 8–12, 32, 33, 58, 59, 74, 78, 99, 101. Polynuclear aromatic hydrocarbons[18]	G, FP-lined cap	Cool, ≤6 °C[9], store in dark, 0.008% $Na_2S_2O_3$ [5]	7 days until extraction, 40 days after extraction
15, 16, 21, 31, 87. Haloethers[18]	G, FP-lined cap	Cool, ≤6 °C[9], 0.008% $Na_2S_2O_3$ [5]	7 days until extraction, 40 days after extraction
29, 35–37, 63–65, 107. Chlorinated hydrocarbons[18]	G, FP-lined cap	Cool, ≤6 °C[9]	7 days until extraction, 40 days after extraction
60–62, 66–72, 85, 86, 95–97, 102, 103. CDDs/CDFs[18]			
Aqueous Samples: Field and Lab Preservation	G	Cool, ≤6 °C[9], 0.008% $Na_2S_2O_3$ [5], pH<9	1 year

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Parameter No./Name (refers to parameter number on Tables IA,B, C, D,E, F, G & H as noted)	Container[1]	Preservation[2, 3]	Maximum holding time[4]
Solids and Mixed-Phase Samples: Field Preservation	G	Cool, ≤6 °C[9]	7 days
Tissue Samples: Field Preservation	G	Cool, ≤6 °C[9]	24 hours
Solids, Mixed-Phase, and Tissue Samples: Lab Preservation	G	Freeze, ≤-10 °C	1 year
Table ID—Pesticides			
Tests: 1–70. Pesticides[18]	G, FP-lined cap	Cool, ≤6 °C[9], pH 5–9[22]	7 days until extraction, 40 days after extraction
Table IE—Radiological Tests:			
1–5. Alpha, beta, and radium	P, FP, G	HNO_3 to pH<2	6 months
Table IH—Bacterial Tests:			
1. *E. coli*			
2. Enterococci	PA, G	Cool, <10 °C, 0.008% $Na_2S_2O_3$ [5]	6 hours[6]
Table IH—Protozoan Tests:			
8. Cryptosporidium	LDPE; field filtration	0–8 °C	96 hours.[23]
9. Giardia	LDPE; field filtration	0–8 °C	96 hours[23]

Reference: This table is adapted from Table II, 40 CFR Part 136, 2007

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[1] ‘‘P’’ is polyethylene; ‘‘FP’’ is fluoropolymer (polytetrafluoroethylene (PTFE; Teflon®), or other fluoropolymer, unless stated otherwise in this Table II; ‘‘G’’ is glass; ‘‘PA’’ is any plastic that is made of a sterlizable material (polypropylene or other autoclavable plastic); ‘‘LDPE’’ is low density polyethylene.

[2] Except where noted in this Table II and the method for the parameter, preserve each grab sample within 15 minutes of collection. For a composite sample collected with an automated sampler (e.g., using a 24-hour composite sampler; see 40 CFR 122.21(g)(7)(i) or 40 CFR Part 403, Appendix E), refrigerate the sample at ≤6 °C during collection unless specified otherwise in this Table II or in the method(s). For a composite sample to be split into separate aliquots for preservation and/or analysis, maintain the sample at ≤6 °C, unless specified otherwise in this Table II or in the method(s), until collection, splitting, and preservation is completed. Add the preservative to the sample container prior to sample collection when the preservative will not compromise the integrity of a grab sample, a composite sample, or an aliquot split from a composite sample; otherwise, preserve the grab sample, composite sample,

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or aliquot split from a composite sample within 15 minutes of collection. If a composite measurement is required but a composite sample would compromise sample integrity, individual grab samples must be collected at prescribed time intervals (e.g., 4 samples over the course of a day, at 6-hour intervals). Grab samples must be analyzed separately and the concentrations averaged. Alternatively, grab samples may be collected in the field and composited in the laboratory if the compositing procedure produces results equivalent to results produced by arithmetic averaging of the results of analysis of individual grab samples. For examples of laboratory compositing procedures, see EPA Method 1664A (oil and grease) and the procedures at 40 CFR 141.34(f)(14)(iv) and (v) (volatile organics).

[3] When any sample is to be shipped by common carrier or sent via the U.S. Postal Service, it must comply with the Department of Transportation Hazardous Materials Regulations (49 CFR Part 172). The person offering such material for transportation is responsible for ensuring such compliance. For the preservation requirements of Table II, the Office of Hazardous Materials, Materials Transportation Bureau, Department of Transportation has determined that the Hazardous Materials Regulations do not apply to the following materials: Hydrochloric acid (HCl) in water solutions at concentrations of 0.04% by weight or less (pH about 1.96 or greater); Nitric acid (HNO3) in water solutions at concentrations of 0.15% by weight or less (pH about 1.62 or greater); Sulfuric acid (H2SO4) in water solutions at concentrations of 0.35% by weight or less (pH about 1.15 or greater); and Sodium hydroxide (NaOH) in water solutions at concentrations of 0.080% by weight or less (pH about 12.30 or less).

[4] Samples should be analyzed as soon as possible after collection. The times listed are the maximum times that samples may be held before the start of analysis and still be considered valid (e.g., samples analyzed for fecal coliforms may be held up to 6 hours prior to commencing analysis). Samples may be held for longer periods only if the permittee or monitoring laboratory has data on file to show that, for the specific types of samples under study, the analytes are stable for the longer time, and has received a variance from the Regional Administrator under § 136.3(e). For a grab sample, the holding time begins at the time of collection. For a composite sample collected with an automated sampler (e.g., using a 24-hour composite sampler; see 40 CFR 122.21(g)(7)(i) or 40 CFR Part 403, Appendix E), the holding time begins at the time of the end of collection of the composite sample. For a set of grab samples composited in the field or laboratory, the holding time begins at the time of collection of the last grab sample in the set. Some samples may not be stable for the maximum time period given in the table. A permittee or monitoring laboratory is obligated to hold the sample for a shorter time if it knows that a shorter time is necessary to maintain sample stability. See § 136.3(e) for details. The date and time of collection of an individual grab sample is the date and time at which the sample is collected. For a set of grab samples to be composited, and that are all collected on the same calendar date, the date of collection is the date on which the samples are collected. For a set of grab samples to be composited, and that are collected across two calendar dates, the date of collection is the dates of the two days; e.g., November 14–15. For a composite sample collected automatically on a given date, the

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date of collection is the date on which the sample is collected. For a composite sample collected automatically, and that is collected across two calendar dates, the date of collection is the dates of the two days; e.g., November 14–15.

[5] Add a reducing agent only if an oxidant (e.g., chlorine) is present. Reducing agents shown to be effective are sodium thiosulfate ($Na_2S_2O_3$), ascorbic acid, sodium arsenite ($NaAsO_2$), or sodium borohydride ($NaBH_4$). However, some of these agents have been shown to produce a positive or negative cyanide bias, depending on other substances in the sample and the analytical method used. Therefore, do not add an excess of reducing agent. Methods recommending ascorbic acid (e.g., EPA Method 335.4) specify adding ascorbic acid crystals, 0.1–0.6 g, until a drop of sample produces no color on potassium iodide (KI) starch paper, then adding 0.06 g (60 mg) for each liter of sample volume. If $NaBH_4$ or $NaAsO_2$ is used, 25 mg/L $NaBH_4$ or 100 mg/L $NaAsO_2$ will reduce more than 50 mg/L of chlorine (see method ''Kelada-01'' and/or Standard Method

4500–CN^- for more information). After adding reducing agent, test the sample using KI paper, a test strip (e.g. for chlorine, SenSafeTM Total Chlorine Water Check 480010) moistened with acetate buffer solution (see Standard Method 4500–Cl.C.3e), or a chlorine/oxidant test method (e.g., EPA Method 330.4 or 330.5), to make sure all oxidant is removed. If oxidant remains, add more reducing agent. Whatever agent is used, it should be tested to assure that cyanide results are not affected adversely.

[6] Samples analysis should begin immediately, preferably within 2 hours of collection. The maximum transport time to the laboratory is 6 hours, and samples should be processed within 2 hours of receipt at the laboratory.

[7] For fecal coliform samples for sewage sludge (biosolids) only, the holding time is extended to 24 hours for the following sample types using either EPA Method 1680 (LTB–EC) or 1681 (A–1): Class A composted, Class B aerobically digested, and Class B anaerobically digested.

[8] Sufficient ice should be placed with the samples in the shipping container to ensure that ice is still present when the samples arrive at the laboratory. However, even if ice is present when the samples arrive, it is necessary to immediately measure the temperature of the samples and confirm that the preservation temperature maximum has not been exceeded. In the isolated cases where it can be documented that this holding temperature cannot be met, the permittee can be given the option of on-site testing or can request a variance. The request for a variance should include supportive data which show that the toxicity of the effluent samples is not reduced because of the increased holding temperature.

[9] Aqueous samples must be preserved at ≤6 °C, and should not be frozen unless data demonstrating that sample freezing does not adversely impact sample integrity is maintained on file and accepted as valid by the regulatory authority. Also, for purposes of NPDES monitoring, the specification of ''≤°C'' is used in place of the ''4 °C'' and ''< 4 °C'' sample temperature requirements listed in some methods. It is not necessary to measure the sample temperature to three significant figures (1/100th of 1 degree); rather, three

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significant figures are specified so that rounding down to 6 °C may not be used to meet the ≤6 °C requirement. The preservation temperature does not apply to samples that are analyzed immediately (less than 15 minutes).

[10] Sample collection and preservation: Collect a volume of sample appropriate to the analytical method in a bottle of the material specified. If the sample can be analyzed within 48 hours and sulfide is not present, adjust the pH to > 12 with sodium hydroxide solution (e.g., 5% w/v), refrigerate as specified, and analyze within 48 hours. Otherwise, to extend the holding time to 14 days and mitigate interferences, treat the sample immediately using any or all of the following techniques, as necessary, followed by adjustment of the sample pH to > 12 and refrigeration as specified. There may be interferences that are not mitigated by approved procedures. Any procedure for removal or suppression of an interference may be employed, provided the laboratory demonstrates that it more accurately measures cyanide. Particulate cyanide (e.g., ferric ferrocyanide) or a strong cyanide complex (e.g., cobalt cyanide) are more accurately measured if the laboratory holds the sample at room temperature and pH > 12 for a minimum of 4 hours prior to analysis, and performs UV digestion or dissolution under alkaline (pH=12) conditions, if necessary.

(1) SULFUR: To remove elemental sulfur (S8), filter the sample immediately. If the filtration time will exceed 15 minutes, use a larger filter or a method that requires a smaller sample volume (e.g., EPA Method 335.4 or Lachat Method 01). Adjust the pH of the filtrate to > 12 with NaOH, refrigerate the filter and filtrate, and ship or transport to the laboratory. In the laboratory, extract the filter with 100 mL of 5% NaOH solution for a minimum of 2 hours. Filter the extract and discard the solids. Combine the 5% NaOH-extracted filtrate with the initial filtrate, lower the pH to approximately 12 with concentrated hydrochloric or sulfuric acid, and analyze the combined filtrate. Because the detection limit for cyanide will be increased by dilution by the filtrate from the solids, test the sample with and without the solids procedure if a low detection limit for cyanide is necessary. Do not use the solids procedure if a higher cyanide concentration is obtained without it. Alternatively, analyze the filtrates from the sample and the solids separately, add the amounts determined (in µg or mg), and divide by the original sample volume to obtain the cyanide concentration.

(2) SULFIDE: If the sample contains sulfide as determined by lead acetate paper, or if sulfide is known or suspected to be present, immediately conduct one of the volatilization treatments or the precipitation treatment as follows: Volatilization—Headspace expelling. In a fume hood or well-ventilated area, transfer 0.75 liter of sample to a 4.4 L collapsible container (e.g., CubitainerTM). Acidify with concentrated hydrochloric acid to pH
< 2. Cap the container and shake vigorously for 30 seconds. Remove the cap and expel the headspace into the fume hood or open area by collapsing the container without expelling the sample. Refill the headspace by expanding the container. Repeat expelling a total of five headspace volumes. Adjust the pH to > 12, refrigerate, and ship or transport to the laboratory. Scaling to a smaller or larger sample volume must maintain the air to sample volume ratio. A larger volume of air will result in too great a loss of cyanide (> 10%). Dynamic stripping: In a fume hood or well- ventilated area, transfer 0.75 liter of sample to a container of the material specified and acidify with concentrated hydrochloric acid to pH < 2. Using a calibrated air sampling pump or flowmeter, purge the acidified sample into the fume hood or open area through a fritted glass aerator at a flow rate of 2.25 L/min for 4 minutes. Adjust the pH to >

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12, refrigerate, and ship or transport to the laboratory. Scaling to a smaller or larger sample volume must maintain the air to sample volume ratio. A larger volume of air will result in too great a loss of cyanide (> 10%). Precipitation: If the sample contains particulate matter that would be removed by filtration, filter the sample prior to treatment to assure that cyanide associated with the particulate matter is included in the measurement. Ship or transport the filter to the laboratory. In the laboratory, extract the filter with 100 mL of 5% NaOH solution for a minimum of 2 hours. Filter the extract and discard the solids. Combine the 5% NaOH-extracted filtrate with the initial filtrate, lower the pH to approximately 12 with concentrated hydrochloric or sulfuric acid, and analyze the combined filtrate. Because the detection limit for cyanide will be increased by dilution by the filtrate from the solids, test the sample with and without the solids procedure if a low detection limit for cyanide is necessary. Do not use the solids procedure if a higher cyanide concentration is obtained without it. Alternatively, analyze the filtrates from the sample and the solids separately, add the amounts determined (in µg or mg), and divide by the original sample volume to obtain the cyanide concentration. For removal of sulfide by precipitation, raise the pH of the sample to > 12 with NaOH solution, then add approximately 1 mg of powdered cadmium chloride for each mL of sample. For example, add approximately 500 mg to a 500-mL sample. Cap and shake the container to mix. Allow the precipitate to settle and test the sample with lead acetate paper. If necessary, add cadmium chloride but avoid adding an excess. Finally, filter through 0.45 micron filter. Cool the sample as specified and ship or transport the filtrate and filter to the laboratory. In the laboratory, extract the filter with 100 mL of 5% NaOH solution for a minimum of 2 hours. Filter the extract and discard the solids. Combine the 5% NaOH-extracted filtrate with the initial filtrate, lower the pH to approximately 12 with concentrated hydrochloric or sulfuric acid, and analyze the combined filtrate. Because the detection limit for cyanide will be increased by dilution by the filtrate from the solids, test the sample with and without the solids procedure if a low detection limit for cyanide is necessary. Do not use the solids procedure if a higher cyanide concentration is obtained without it. Alternatively, analyze the filtrates from the sample and the solids separately, add the amounts determined (in µg or mg), and divide by the original sample volume to obtain the cyanide concentration. If a ligand-exchange method is used (e.g., ASTM D6888), it may be necessary to increase the ligand-exchange reagent to offset any excess of cadmium chloride.

(3) SULFITE, THIOSULFATE, OR THIOCYANATE: If sulfite, thiosulfate, or thiocyanate is known or suspected to be present, use UV digestion with a glass coil (Method Kelada-01) or ligand exchange (Method OIA–1677) to preclude cyanide loss or positive interference.

(4) ALDEHYDE: If formaldehyde, acetaldehyde, or another water-soluble aldehyde is known or suspected to be present, treat the sample with 20 mL of 3.5% ethylenediamine solution per liter of sample.

(5) CARBONATE: Carbonate interference is evidenced by noticeable effervescence upon acidification in the distillation flask, a reduction in the pH of the absorber solution, and incomplete cyanide spike recovery. When significant carbonate is present, adjust the pH to ≥12 using calcium hydroxide instead of sodium hydroxide. Allow the precipitate to settle and decant or filter the sample prior to analysis (also see Standard Method 4500–CN.B.3.d).

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(6) CHLORINE, HYPOCHLORITE, OR OTHER OXIDANT: Treat a sample known or suspected to contain chlorine, hypochlorite, or other oxidant as directed in footnote 5.

[11] For dissolved metals, filter grab samples within 15 minutes of collection and before adding preservatives. For a composite sample collected with an automated sampler (e.g., using a 24-hour composite sampler; see 40 CFR 122.21(g)(7)(i) or 40 CFR Part 403, Appendix E), filter the sample within 15 minutes after completion of collection and before adding preservatives. If it is known or suspected that dissolved sample integrity will be compromised during collection of a composite sample collected automatically over time (e.g., by interchange of a metal between dissolved and suspended forms), collect and filter grab samples to be composited (footnote 2) in place of a composite sample collected automatically.

[12] To achieve the 28-day holding time, use the ammonium sulfate buffer solution specified in EPA Method 218.6. The allowance in this footnote supersedes preservation and holding time requirements in the approved hexavalent chromium methods, unless this supersession would compromise the measurement, in which case requirements in the method must be followed.

[13] Samples collected for the determination of trace level mercury (<100 ng/L) using EPA Method 1631 must be collected in tightly-capped fluoropolymer or glass bottles and preserved with BrCl or HCl solution within 48 hours of sample collection. The time to preservation may be extended to 28 days if a sample is oxidized in the sample bottle. A sample collected for dissolved trace level mercury should be filtered in the laboratory within 24 hours of the time of collection. However, if circumstances preclude overnight shipment, the sample should be filtered in a designated clean area in the field in accordance with procedures given in Method 1669. If sample integrity will not be maintained by shipment to and filtration in the laboratory, the sample must be filtered in a designated clean area in the field within the time period necessary to maintain sample integrity. A sample that has been collected for determination of total or dissolved trace level mercury must be analyzed within 90 days of sample collection.

[14] An aqueous sample may be collected and shipped without acid preservation. However, acid must be added at least 24 hours before analysis to dissolve any metals that adsorb to the container walls. If the sample must be analyzed within 24 hours of collection, add the acid immediately (see footnote 2). Soil and sediment samples do not need to be preserved with acid. The allowances in this footnote supersede the preservation and holding time requirements in the approved metals methods.

[15] Guidance applies to samples to be analyzed by GC, LC, or GC/MS for specific compounds.

[16] If the sample is not adjusted to pH 2, then the sample must be analyzed within seven days of sampling.

[17] The pH adjustment is not required if acrolein will not be measured. Samples for acrolein receiving no pH adjustment must be analyzed within 3 days of sampling.

Table FS1000-4
40 CFR Part 136 TABLE II: Required Containers, Preservation Techniques, and Holding Times
Applicable to **all** Non-Potable Water Samples (includes wastewater, surface water, and groundwater)

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[18] When the extractable analytes of concern fall within a single chemical category, the specified preservative and maximum holding times should be observed for optimum safeguard of sample integrity (i.e., use all necessary preservatives and hold for the shortest time listed). When the analytes of concern fall within two or more chemical categories, the sample may be preserved by cooling to ≤6 °C, reducing residual chlorine with 0.008% sodium thiosulfate, storing in the dark, and adjusting the pH to 6–9; samples preserved in this manner may be held for seven days before extraction and for forty days after extraction. Exceptions to this optional preservation and holding time procedure are noted in footnote 5 (regarding the requirement for thiosulfate reduction), and footnotes 19, 20 (regarding the analysis of benzidine).

[19] If 1,2-diphenylhydrazine is likely to be present, adjust the pH of the sample to 4.0 ± 0.2 to prevent rearrangement to benzidine.

[20] Extracts may be stored up to 30 days at < 0 °C.

[21] For the analysis of diphenylnitrosamine, add 0.008% $Na_2S_2O_3$ and adjust pH to 7–10 with NaOH within 24 hours of sampling

[22] The pH adjustment may be performed upon receipt at the laboratory and may be omitted if the samples are extracted within 72 hours of collection. For the analysis of aldrin, add 0.008% $Na_2S_2O_3$.

[23] Holding time is calculated from time of sample collection to elution for samples shipped to the laboratory in bulk and calculated from the time of sample filtration to elution for samples filtered in the field

Table FS 1000-5
Approved Water and Wastewater Procedures, Containers, Preservation and Holding Times
For Analytes not Found in 40 CFR 136

Analyte	Methods	Reference [1]	Container[2]	Preservation[3]	Maximum Holding Time[4]
Bromine	DPD Colorimetric[5]	SM 4500-Cl-G	P, G	None required	Analyze immediately
Bromates	Ion Chromatography	EPA 300.0 [6]	P, G	Cool 4°C	30 days
Chlorophylls	Spectrophotometric	SM 10200 H	P, G[7]	Dark 4°C Filtered, dark, ⁻20°C	48 hours chilled until filtration[8], and analyze immediately or 48 hours chilled until filtration[8],and 28 days (frozen)after filtration
Corrosivity	Calculated ($CaCO_3$ Stability, Langelier Index)	SM 2330 ASTM D513-92	P, G	Cool 4°C[9]	7 days[9]
FL-PRO	Gas Chromatography	DEP (11/1/95)	G only	Cool 4°C, H_2SO_4 or HCl to pH<2	7 days until extraction, 40 days after extraction
Odor	Human Panel	SM 2150	G only	Cool 4°C	6 hours
Salinity	Electrometric [10] Hydrometric[10]	SM 2520 B SM 2520 C	G, wax seal	Analyze immediately or use wax seal	30 days[10]
Taste	Human Panel	SM 2160 B, C, D ASTM E679-91	G only	Cool 4°C	24 hours
Total Dissolved Gases	Direct-sensing Membrane-diffusion	SM 2810	_____	_____	Analyze in-situ
Total Petroleum Hydrocarbons	Gravimetry	EPA 1664	G only	Cool 4°C, H_2SO_4 or HCl to pH<2	28 days
Transparency	Irradiometric[11]	62-302.200(6), FAC	_____	_____	Analyze in-situ
Un-ionized Ammonia	Calculated[12]	DEP-SOP[13]	P, G	Cool 4°C $Na_2S_2O_3$ [12]	8 hours unpreserved 28 days preserved [12]
Organic Pesticides[14]	GC and HPLC	EPA (600-series) [14]	[15]	[15]	[15]

[1] SM XXXX = procedures from "Standard Methods for the Examination of Water and Wastewater", APHA-AWWA-WPCF, 20th edition, 1998 and Standard Methods Online.

ASTM XXXX-YY = procedure from "Annual Book of ASTM Standards", Volumes 11.01 and 11.02 (Water I and II), 1999.

[2] P = plastic, G = glass.

[3] When specified, sample preservation should be performed immediately upon sample collection.

[4] The times listed are the maximum times that samples may be held before analysis and still be considered valid.

Table FS 1000-5
Approved Water and Wastewater Procedures, Containers, Preservation and Holding Times
For Analytes not Found in 40 CFR 136

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[5] The approved procedure is for residual chlorine. However, in the absence of chlorine, the DPD colorimetric procedure can be adapted to measure bromine content of the sample. In such case, the validity of this assumption must be verified by using another procedure for chlorine which is not affected by the presence of bromine (i.e., negligible interference).

[6] The Determination of Inorganic Anions in Water by Ion Chromatography", EPA Method 300.0, Revised August 1993, by John D. Pfaff, U. S. EPA Cincinnati, Ohio 45268.

[7] Collect samples in opaque bottles and process under reduced light.

[8] Samples must be filtered within 48 hours of collection. Add magnesium carbonate to the filter while the last of the sample passes through the filter..

[9] Temperature and pH must be measured on site at the time of sample collection. 7 days is the maximum time for laboratory analysis of total alkalinity, calcium ion and total solids.

[10] The electrometric and hydrometric analytical methods are suited for field use. The argentometric method is suited for laboratory use. Samples collected for laboratory analysis, when properly sealed with paraffin waxed stopper, may be held indefinitely. The maximum holding time of 30 days is recommended as a practical regulatory limit.

[11] Transparency in surface waters is defined as a compensation point for photosynthetic activity, i.e., the depth at which one percent of the light intensity entering at the water surface remains unabsorbed. The DEP Chapter 62-302, FAC requires that the light intensities at the surface and subsurface be measured simultaneously by irradiance meters such as the Kahlsico Underwater Irradiometer, Model No. 268 WA 310, or an equivalent device having a comparable spectral response.

[12] The results of the measurements of pH, temperature, salinity (if applicable) and the ammonium ion concentration in the sample are used to calculate the concentration of ammonia in the unionized state. Temperature, pH and salinity must be measured on-site at the time of sample collection. Laboratory analysis of the ammonium ion concentration should be conducted within eight hours of sample collection. If prompt analysis of ammonia is impossible, preserve samples with H_2SO_4 to pH between 1.5 and 2. Acid-preserved samples, stored at 4°C, may be held up to 28 days for ammonia determination. Sodium thiosulfate should only be used if fresh samples contain residual chlorine.

[13] DEP Central Analytical Laboratory, Tallahassee, FL, Revision No. 2, 2-12-2001. The document is available from the DEP Standards & Assessment Section..

[14] Other pesticides listed in approved EPA methods (608.1, 608.2, 614, 614.1, 615, 617, 618, 619, 622, 622.1, 627, 629, 631, 632, 632.1, 633, 642, 643, 644 and 645) that are not included in Table ID of 40 CFR Part 136 (July 2007).

[15] Container, preservation and holding time as specified in each individual method must be followed.

Table FS 1000-6
Recommended Sample Containers, Sample Volumes, Preservation Techniques and Holding Times for Residuals, Soil and Sediment Samples

Analyte	Methods	References	Container	Preservation	Maximum Holding Times
Volatile Organics	Purge-and-Trap GC and GC-MS	8015, 8260, 8021, 5035	See Table 1000-7		
Semivolatile Organics	GC, HPLC, and GC-MS	8041, 8061, 8070, 8081, 8082, 8091, 8111, 8121, 8131, 8141, 8151, 8270, 8275, 8280, 8290, 8310, 8315, 8316, 8318, 8321, 8325, 8330, 8331, 8332, 8410, 8430, 8440, FL-PRO	Glass, 8 oz widemouth with Teflon® -Lined lid	Cool 4°C [1]	14 days until extraction, 40 days after extraction
Dioxins		8290	Amber Glass, 8 oz widemouth with Teflon® -Lined lid	Cool 4°C[1] in dark	30 days until extraction, 45 days after extraction
Total Metals-except mercury and chromium VI methods	Flame AA, Furnace AA, Hydride and ICP	All 7000-series (except 7195, 7196, 7197, 7198, 7470 and 7471), and 6010 (ICP)	Glass or plastic 8 oz widemouth (200 grams sample)	None	6 months
Chromium VI	Colorimetric, Chelation with Flame AA (200 gram sample)	7196 and 7197 (prep 3060)	Glass or plastic, 8 oz widemouth (200 gram sample)	Cool 4°± 2°C[1]	1 month until extraction, 4 days after extraction [2]
Mercury	Manual Cold Vapor AA	7471	Glass or plastic 8 oz widemouth (200 grams sample)	Cool 4°± 2°C[1]	28 days
Microbiology (MPN)		MPN	Sterile glass or plastic	Cool 4°C[1]	24 hours
Aggregate Properties			Glass or plastic	Cool 4°C[1]	14 days
Inorganic nonmetallics all except:			Glass or plastic	Cool 4°C[1]	28 days
Sulfite, Nitrate, Nitrite & o-phosphate			Glass or plastic		48 hours
Elemental Phosphorus			Glass		48 hours

Table FS 1000-6
Recommended Sample Containers, Sample Volumes, Preservation Techniques and Holding Times for Residuals, Soil and Sediment Samples

The term “residuals” include: (1) sludges of domestic origin having no specific requirements in Tables FS-1000-4 or FS-1000-9; (2) sludges of industrial origin; and (3) concentrated waste samples.

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[1] Keep soils, sediments and sludges cool at 4°C from collection time until analysis. No preservation is required for concentrated waste samples.

[2] Storage Temperature is 4°C, ±2°C

Table FS 1000-7

Sample Handling, Preservation and Holding Time Table for SW 846 Method 5035

			Sample Container					
Conc. Level	Sampling Device	Collection Procedure	Type	Vial Preparation	Preservation	Sample Preparation	Max HT[①]	Determinative Procedure
□200 ug/kg	Coring Device	5035 - Section 6.2.1	Glass Vial w/ PTFE-silicone Septum	5035 - 6.1.1	$NaHSO_4$ / 4ºC	5035 - Section 7.2	14 D	Any recognized VOC Method
				5035 - 6.1.1[②]	4ºC	5035 - Section 7.2	48 H	Any recognized VOC Method
				5035 - 6.1.1[②]	4ºC / -10ºC[③,④]	5035 - Section 7.2	48 H / 14 D[⑤]	Any recognized VOC Method
	EnCore or equivalent	5035 - Section 6.2.1	EnCore or equivalent	5035 - 6.1.1[②,⑥,⑦]	4ºC	5035 - Section 7.2	48 H	Any recognized VOC Method
		5035 - Section 6.2.1	EnCore or equivalent	5035 - 6.1.1[⑥,⑦]	$NaHSO_4$ / 4ºC	5035 - Section 7.2[⑤]	48 H / 14 D[⑤]	Any recognized VOC Method
		5035 - Section 6.2.1	EnCore or equivalent	5035-6.1.1[②⑥⑦]	4ºC / -10ºC[③,④]	5035 - Section 7.2[⑤]	48 H / 14 D[⑤]	Any recognized VOC Method
□200 ug/kg	EnCore or equivalent	5035 - Section 6.2.2.3[⑥]	EnCore or equivalent	5035 - 6.1.3[⑥,⑦]	4ºC	5035 - Sections 7.3.2 & 7.3.3[⑤]	48 H / 14 D[⑤]	Any recognized VOC Method
□200 ug/kg[⑧]	Coring Device	5035 - Section 6.2.2.3[⑨]	Glass Vial w/ PTFE-silicone Septum	6.1.3[⑨]	Methanol/PEG + 4ºC	5035 - Section 7.3.4	14 D	Any recognized VOC Method
	Conventional Devices	DEP SOP - Section 4.3	Glass w/ PTFE-silicone Septum	6.1.2	4ºC	5035 - Sections 7.3.1 - 7.3.3	14 D	Any recognized VOC Method
Oily Waste	Conventional Devices	5035 - Section 6.2.4.2	Glass w/ PTFE-silicone Septum	6.1.4	4ºC	5035 - Sections 7.4.1 - 7.4.2	14 D	Any recognized VOC Method
	Conventional Devices	5035 - Section 6.2.4.1	Glass w/ PTFE-silicone Septum	6.1.4	Methanol/PEG + 4ºC	5035 - Sections 7.4.3	14 D	Any recognized VOC Method
Dry Wt.	Conventional Devices		Glass with Teflon liner		4ºC	5035 - Section 7.5		
Soil Screen	Conventional Devices	DEP SOP - Section 4.3	Glass w/ PTFE-silicone Septum		4ºC	5035 - Section 7.1	14 D	Any recognized VOC Method

Table FS 1000-7

Sample Handling, Preservation and Holding Time Table for SW 846 Method 5035

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① Maximum time allowable from time/date of collection to sample analysis.

② Eliminate 6.1.1.2; use only organic-free water.

③ Contents of sampling device must be transported to the laboratory at 4ºC and stored at -10ºC.

④ In order to ensure that vials do not break during freezing, they should be stored on their side or at a slanted angle to maximize surface area.

⑤ Maximum allowable time at 4ºC is 48 hours; maximum allowable time to sample analysis is 14 days (from time of sample collection).

⑥ Conducted in the laboratory.

⑦ Entire contents of sampling device are extruded into the sample analysis vial containing the appropriate solvent.

⑧ Procedures are limited only to those situations or programs in which the maximum contamination level does not exceed 200 ug/kg.

⑨ Methanolic preservation in the field is not recommended, but may be used if approved by an DEP program.

FS 1000-8
Preservation Methods and Holding Times for Drinking Water Samples that Differ from 40 CFR Part 136, Table II

Analyte	Preservation[1]	Holding Time[2]	Holding Time for Extract[3]	Container[4]
MICROBIOLOGICAL-BACTERIA	Cool < 10°C, $Na_2S_2O_3$[5]			P or G
Total Coliforms, fecal coliforms & *E. coli* in drinking water	Cool < 10°C[6], $Na_2S_2O_3$[5]	30 Hours[7]		P or G
Total coliforms and fecal coliforms in source water Heterotrophic bacteria in drinking water	Cool < 10°C, $Na_2S_2O_3$[5]	8 hours		P or G
Gross Alpha	Conc. HCl or HNO_3 to pH <2 [8,9]	6 mo		P or G
Gross beta	Conc. HCl or HNO to pH <2[8,9]	6 mo		P or G
Strontium-89	Conc. HCl or HNO to pH <2[8,9]	6 mo		P or G
Strontium-90	Conc. HCl or HNO to pH <2[8,9]	6 mo		P or G
Radium-226	Conc. HCl or HNO to pH <2[8,9]	6 mo		P or G
Radium-228	Conc. HCl or HNO to pH <2[8,9]	6 mo		P or G
Cesium-134	Concentrated HCl to pH <<2[8,9]	6 mo		P or G
Iodine-131	None	8 da		P or G
Tritium	None	6 months		G
Uranium	Conc. HCl or HNO_3 to pH <2[8,9]	6 mo		P or G
Photon emitters	Conc. HCl or HNO_3 to pH <2[8,9]	6 mo		P or G
Asbestos	Cool 4°C	48 hours		P or G
Bromate	Ethylenediamine (50mg/L)	28 days		P or G
Cyanide	Cool, 4C, Ascorbic acid (if chlorinated), NaOH pH>12	14 days		P or G
Nitrate	Cool, 4°C	48 hours		P or G
Nitrate (chlorinated source)	Cool, 4°C	14 days		P or G
Odor	Cool 4°C	24 hours		G
502.2	Sodium Thiosulfate or Ascorbic Acid, 4°C HCl pH<2 if Ascorbic Acid is used	14 days		Glass with PFTE Lined Septum

FS 1000-8
Preservation Methods and Holding Times for Drinking Water Samples that Differ from 40 CFR Part 136, Table II

Analyte	Preservation[1]	Holding Time[2]	Holding Time for Extract[3]	Container[4]
504.1	Sodium Thiosulfate Cool, 4°C,	14 days	4°C, 24 hours	Glass with PFTE-Lined Septum
505	Sodium Thiosulfate Cool, 4°C	14 days (7 days for Heptachlor)	4°C, 24 hours	Glass with PFTE-Lined Septum
506	Sodium Thiosulfate Cool, 4°C, Dark	14 days	4°C, dark, 14 days	Amber Glass with PFTE-lined Cap
507	Sodium Thiosulfate Cool, 4°C, Dark	14 days (see method for exceptions)	4°C, dark, 14 days	Amber Glass with PFTE-lined Cap
508	Sodium Thiosulfate Cool, 4°C, Dark	7 days (see method for exceptions)	4°C, dark, 14 days	Glass with PFTE-lined Cap
508A	Cool, 4°C	14 days	30 days	Glass with PFTE-lined Cap
508.1	Sodium Sulfite, HCl pH<2, Cool, 4°C	14 days (see method for exceptions)	30 days	Glass with PFTE-lined Cap
515.1	Sodium Thiosulfate Cool, 4°C, Dark	14 days	4°C, dark, 28 days	Amber Glass with PFTE-lined Cap
515.2	Sodium Thiosulfate HCl pH<2, Cool, 4°C, Dark	14 days	≤ 4°C, dark, 14 days	Amber Glass with PFTE-lined Cap
515.3	Sodium Thiosulfate HCl pH<2, Cool, 4°C, Dark	14 days	≤ 4°C, dark, 14 days	Amber Glass with PFTE-lined Cap
515.4	Sodium Sulfite, HCl pH<2, Cool, ≤10°C for first 48 hours ≤6°C thereafter, Dark	14 days	≤0°C, 21 days	
524.2	Ascorbic Acid, HCl pH<2, Cool 4°C	14 days		Glass with PFTE-lined Septum
525.2	Sodium Sulfite, Dark, Cool, 4°C, HCl pH<2	14 days (see method for exceptions)	≤ 4°C, 30 days from collection	Amber Glass with PFTE-lined Cap
531.1, 6610	Sodium Thiosulfate Monochloroacetic acid, pH<3, Cool, 4°C	Cool 4°C, 28 days		Glass with PFTE-lined Septum
531.2	Sodium Thiosulfate, Potassium Dihydrogen Citrate buffer to pH 4,	28 days		

FS 1000-8
Preservation Methods and Holding Times for Drinking Water Samples that Differ from 40 CFR Part 136, Table II

Analyte	Preservation[1]	Holding Time[2]	Holding Time for Extract[3]	Container[4]
	dark, ≤10°C for first 48 hr, ≤6°C thereafter			
547	Sodium Thiosulfate Cool, 4°C	14 days (18 mo. frozen)		Glass with PFTE-lined Septum
548.1	Sodium Thiosulfate (HCl pH 1.5-2 if high biological activity), Cool, 4°C, Dark	7 days	≤4°C 14 days	Amber Glass with PFTE-lined Septum
549.2	Sodium Thiosulfate (H_2SO_4 pH<2 if biologically active), Cool, 4°C, Dark	7 days	21 days	High Density Amber Plastic or Silanized Amber Glass
550, 550.1	Sodium Thiosulfate Cool, 4°C, HCl pH<2	7 days	550, 30 days 550.1, 40 days Dark, 4°C	Amber Glass with PFTE-lined Cap
551.1	Sodium Thiosulfate, Sodium Sulfite, Ammonium Chloride, pH 4.5-5.0 with phosphate buffer, Cool, 4°C	14 days		Glass with PFTE-lined Septum
552.1	Ammonium chloride, Cool, 4°C, Dark	14 days	≤4°C, dark 48 hours	Amber Glass with PFTE-lined cap
552.2	Ammonium chloride, Cool, 4°C, Dark	14 days	≤4°C, dark 7 days ≤-10°C 14 days	Amber Glass with PFTE-lined cap
555	Sodium Sulfite, HCl, pH ≤ 2, Dark, Cool 4°C	14 days		Glass with PFTE-lined cap
1613B	Sodium Thiosulfate, Cool, 0-4°C, Dark		Recommend 40 days	Amber Glass with PFTE-lined Cap

[1] Preservation, when required, must be done immediately upon sample collection.

[2] Stated values are the maximum regulatory holding times. Sample processing must begin by the stated time.

[3] Stated time is the maximum time a prepared sample extract may be held before analysis.

[4] (P) polyethylene or (G) or glass. For microbiology, plastic sample containers must be made of sterilizable materials (poly-propylene or other autoclavable plastic).

[5] Addition of sodium thiosulfate is only required if the sample has a detectable amount of residual chlorine, as indicated by a field test using EPA Method 330.4 or 330.2 or equivalent.

FS 1000-8
Preservation Methods and Holding Times for Drinking Water Samples that Differ from 40 CFR Part 136, Table II

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[6] Temperature requirement applies only to source water samples, however once received by the laboratory, if sample processing does not begin on the same working day, samples must be refrigerated.

[7] If samples are analyzed after 30 hours, but within 48 hours of collection, the laboratory is to indicate in the analytical report that the data may be invalid because of excessive delay in sample processing. No samples received after 48 hours are to be accepted or analyzed for compliance with the regulations of the Department of Environmental Protection or the Department of Health.

[8] It is recommended that the preservative be added at the time of collection unless suspended solids activity is to be measured. It is also recommended that samples be filtered, if suspended or settleable solids are present, prior to adding preservative, at the time of collection. However, if the sample has to be shipped to a laboratory or storage area, acidification of the sample (in its original container) may be delayed for a period not to exceed 5 days. A minimum of 16 hours must elapse between acidification and analysis.

[9] If HCl is used to acidify samples, which are to be analyzed for gross alpha or gross beta activities, the acid salts must be converted to nitrate salts before transfer of the samples to planchets.

Table FS 1000-9
Containers, Preservation and Holding Times for Biosolids Samples and Protozoans

ANALYTE NAME	*CONTAINER*	*PRESERVATION*	*MAX HOLDING TIME*
Fecal Coliform	Plastic or Glass	Cool 4°C	24 hours
Salmonella	Plastic or Glass	< 10°C	24 hours
Enteric Viruses	Plastic or Glass	Up to 25°C	2 hours
Enteric Viruses	Plastic or Glass	2 to 10°C	48 hours
Specific Oxygen Uptake Rate	Plastic or Glass	None	As Soon As Possible
Helminth OVA	Plastic or Glass	< 4°C (Do not Freeze)	24 hours
Cryptosporidium/Giardia	Plastic or Glass	0 - 8°C (Do not Freeze)*	96 Hours
Total Solids	Plastic or Glass	≤6°C (Do not Freeze)	7 days
Metallics	Plastic or Glass	See Tables FS 1000-4, FS 1000-5 and FS 1000-6	
Other Inorganic Pollutants	Plastic or Glass	See Tables FS 1000-4, FS 1000-5 and FS 1000-6	

***Dechlorinate bulk samples when applicable**

Table FS 1000-10
Container Materials, Preservation, and Holding Times for Fish and Shellfish

Analyte	Matrix	Sample Container	Field (Transport to Lab)		Laboratory	
			Preservation	Maximum Shipping Time	Storage	Holding Time
	Whole Organism (Fish, shellfish, etc.	Foil-wrap each organism (or composite for shellfish) and transport in waterproof plastic bag				
Mercury	Tissue (fillets and edible portions, homogenates)	Plastic, borosilicate glass, quartz, PTFE			Freeze at <-20°C	28 days
Other metals	Tissue (fillets and edible portions, homogenates)	Plastic, borosilicate glass, quartz, PTFE	Cool in wet ice or:	24 hours	Freeze at <-20°C	6 months
Organics	Tissue (fillets and edible portions, homogenates)	Borosilicate glass, PTFE, quartz, aluminum foil	Freeze on dry ice	48 hours	Freeze at <-20°C	1 year
Dioxin	Tissue (fillets and edible portions, homogenates)	Amber containers: Borosilicate glass, PTFE, quartz, aluminum foil			Freeze at <-20°C	30 days until extraction, 15 days after extraction
Lipids	Tissue (fillets and edible portions, homogenates)	Plastic, borosilicate glass, quartz, PTFE			Freeze at <-20°C	1 year

PTFE = Polytetrafluoroethylene (Teflon)

Table FS 1000-11
Holding Times for SPLP or TCLP Extraction, Sample Preparation and Determinative Analysis

Holding Time (Days)				
	From: Field Collection To: SPLP or TCLP Extraction	From: SPLP or TCLP Extraction To: Preparative Extraction	From: Preparative Extraction To: Determinative Analysis	Total Elapsed Time
Volatiles	14	NA	14	28
Semi-Volatiles	14	7	40	61
Mercury	28	NA	28	56
Metals, except Mercury	180	NA	180	360

NA – Not Applicable

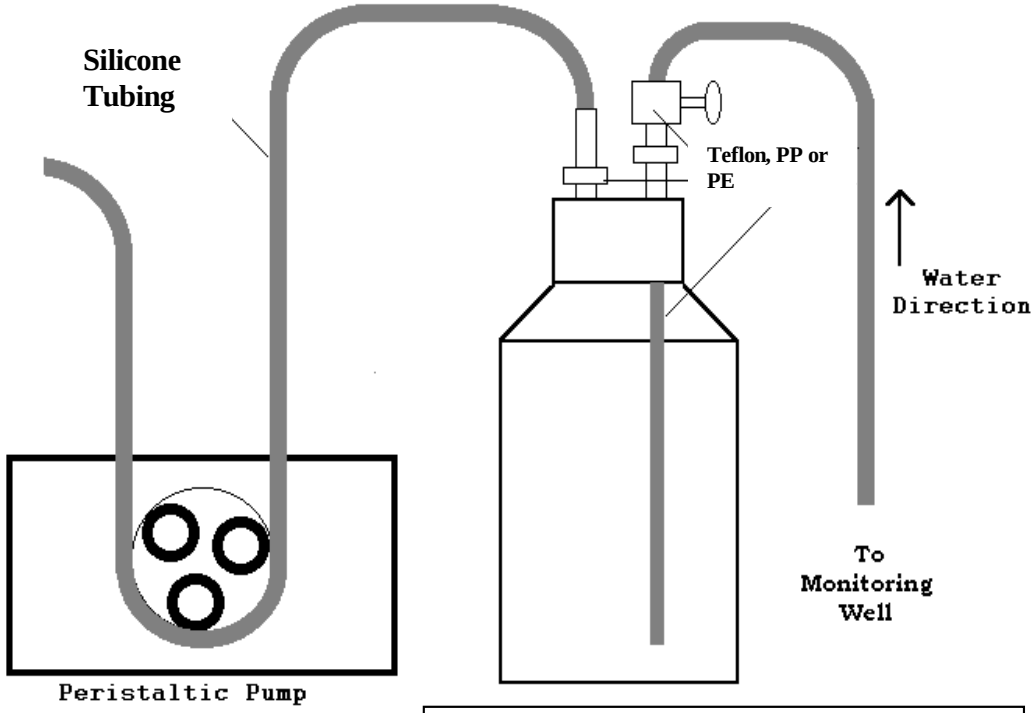
Table FS 1000-12
Preventive Maintenance Tasks

INSTRUMENT/ACTIVITY	FREQUENCY
REFRIGERATORS, INCUBATORS, OVENS	
Clean interior	Monthly
Check thermometer temperature against certified thermometer or equivalent	Annually
ANYTICAL BALANCES	
Clean pan and compartment	Daily[1]
Check with Class S weights	Monthly
Manufacturer cleaning and calibration	Annually
pH AND ION SELECTIVE ELECTRODES	
PROBE	
Check probe for cracks and proper levels of filling solution; check reference junction; clean electrode	Daily, Replace as necessary
Check response time	Daily[1]
METER	
Check batteries and electronics for loose connections and cracked leads	Daily[1], Replace as necessary
TURBIDIMETER	
Clean instrument housing	Monthly
Clean cells	Daily[1]
CONDUCTIVITY METER	
Check batteries and probe cables	Daily[1]
Replatinize Probe	Per manufacturer's recommendations
DISSOLVED OXYGEN METERS	
PROBE	
Check membrane for deterioration; check filling solution	Daily[1], Replace as necessary
METER	
Battery level and electronics checked	Daily[1], Replace as necessary
THERMOMETERS	
Check for cracks and gaps in the mercury	Daily[1], Replace as necessary
TEMPERATURE PROBE	
Check connections, cables	Daily[1]
Check against calibrated thermometer	Daily[1]
AUTOMATIC SAMPLE COLLECTION SYSTEMS (e.g., ISCO, Sigma)	
Check sampler operation (forward, reverse, automatic through three cycles of the purge-pump-purge cycle)	Daily[1]Prior to Sampling Event
Check purge-pump-purge cycle when sampler is installed	Daily[1]Prior to Sampling Event
Check the flow pacer that activates the sampler to assure proper operation	Daily[1]Prior to Sampling Event
Check desiccant	Daily[1], Replace as Necessary
Check batteries	Daily[1], Replace as Necessary
Check pumping rate against manufacturer's specifications	Daily[1], Replace as Necessary

[1]Daily is defined as prior to use or a 12-hour period if equipment is run continuously

Figure FS 1000-1

Organic Trap Configuration for Collecting Extractable Organics with a Peristaltic Pump



FS 2000. GENERAL AQUEOUS SAMPLING

See also the following Standard Operating Procedures:

- FA 1000 Administrative Procedures
- FC 1000 Cleaning/Decontamination Procedures
- FD 1000-9000 Documentation Procedures
- FM 1000 Field Planning and Mobilization
- FQ 1000 Field Quality Control Requirements

1. COMMON PROCEDURES

The following procedures are applicable to the collection of all water samples.

1.1. Refer to FS 1000 for procedures that are common to all types of sample collection including general preservation and thermal preservation procedures.

1.2. Grab Samples

1.2.1.1. This is an individual sample collected over a period of time, usually all in one motion, generally not exceeding 15 minutes. The 15-minute time limit applies to aqueous samples only. No time limit applies to the collection of solid samples (e.g., residuals).

1.2.1.2. Grab samples represent the conditions that exist at the moment the sample is collected and do not necessarily represent conditions at any other time. Grab sampling is the preferred method of sampling under the following conditions:

- A snapshot of the water quality at a particular instant in time is desired.
- The water or wastewater stream is not continuous (e.g., batch discharges or intermittent flow).
- The characteristics of the water or waste stream are known to be constant or nearly so.
- When conditions are relatively constant over the period of discharge. In lieu of complex sampling activities, a grab sample provides a simple and accurate method of establishing waste characteristics.
- The sample is to be analyzed for analytes whose characteristics are likely to change significantly with time (e.g., dissolved gases, microbiological tests, pH).
- The sample is to be collected for analytes such as Oil and Grease, bacteriological tests or other parameters listed in number 3 of this section where the compositing process could significantly affect the actual concentration.
- Data on maximum/minimum concentrations are desired for a continuous water or wastewater stream.
- When identifying and tracking slug loads and spills.

1.2.1.3. If required, measure the following parameters on grab samples or in-situ.

NOTE: If the permit specifies a composite sample for any of the parameters mentioned below, **FOLLOW THE PERMIT CONDITIONS**

Cyanide	Oil and Grease
Residual Chlorine	pH
Dissolved constituents in field-filtered samples (ortho-phosphorus, metals, etc.)	Specific Conductance
Dissolved Oxygen and other dissolved gases	Un-ionized Ammonia
Microbiological Parameters	Volatile Organic Compounds
TRPHs	Temperature
Total Phenols	

1.3. Composite Samples

1.3.1. A composite sample is a sample collected over time, formed either by continuous sampling or by mixing discrete samples. Composite samples reflect the average characteristics during the compositing period.

1.3.2. Composite samples are used when stipulated in a permit or when:

- The water or wastewater stream is continuous;
- Analytical capabilities are limited;
- Determining average pollutant concentration during the compositing period;
- Calculating mass/unit time loadings; or
- Associating average flow data to parameter concentrations

1.3.3. Composite samples may be collected individually at equal time intervals if the flow rate of the sample stream does not vary more than plus or minus ten percent of the average flow rate or they may be collected proportional to the flow rate. The permit or work plan will specify which composite sample type to use, either time composites or flow proportional composites. The compositing methods, all of which depend on either continuous or periodic sampling, are described in the following discussions.

1.3.3.1. Time Composite Sample: Time composite samples are based on a constant time interval between samples. A time composite sample can be collected manually or with an automatic sampler. This type of composite is composed of discrete sample aliquots collected in one container at constant time intervals. This method provides representative samples when the flow of the sampled wastewater stream is constant. This type of sample is similar to a sequential composite sample described in number 3.3 of this section.

1.3.3.2. Flow Proportional Composite Sample: Flow proportional samples can be collected automatically with an automatic sampler and a compatible pacing flow measuring device, semi-automatically with a flow chart and an automatic sampler capable of collecting discrete samples, or manually. There are two methods used to collect this type of sample:

- Method 1: Collect a constant sample volume per stream flow (e.g., a 200 mL sample collected for every 5,000 gallons of stream flow) at time intervals proportional to stream flow. This method provides representative samples of all waste streams when the flow is measured accurately.

- Method 2: Collect a sample by increasing the volume of each aliquot as the flow increases, while maintaining a constant time interval between the aliquots (e.g., hourly samples are taken with the sample volume being proportional to the flow at the time the sample is taken).

1.3.3.3. Sequential Composite Sample: Sequential composite samples are composed of discrete samples taken into individual containers at constant time intervals or constant discharge increments. For example, samples collected every 15 minutes are composited for each hour.

- The 24-hour composite is made up from the individual one-hour composites. Each of the 24 individual samples is manually flow-proportioned according to the flow recorded for the hour that the sample represents. Each flow-proportioned sample is then added to the composite samples. The actual compositing of the samples is done by hand and may be done in the field or the laboratory. In most cases, compositing in the field is preferable since only one sample container must be cooled, and then transported to, and handled, in the laboratory. A 24-hour composite is frequently used since an automatic sampler can easily collect the individual samples.

- A variation of the 24-hour composite is to collect a constant volume of sample taken at constant discharge increments, which are measured with a totalizer. For example, one aliquot is collected for every 10,000 gallons of flow

- Sequential sampling is useful to characterize the waste stream because you can determine the variability of the wastewater constituents over a daily period. For example, for pretreatment studies you can visually determine when high strength wastes are being discharged from a facility or when heavy solid loads are being discharged during a 24-hour cycle. You can measure the pH throughout the day. The value of this type of sampling must be weighed against the manpower constraints and sampling goals

1.3.3.4. Continuous Composite Sample: Collected continuously from the stream. The sample may be a constant volume that is similar to the time composite, or the volume may vary in proportion to the flow rate of the waste stream, in which case the sample is similar to the flow proportional composite.

1.3.3.5. Areal Composite: A sample composited from individual grab samples collected on an areal or cross-sectional basis. Areal composites must be made up of equal volumes of grab samples; each grab sample must be collected in an identical manner. Examples include residual samples from grid system points on a land application site, water samples collected at various depths at the same point or from quarter points in a stream, etc sample is similar to the flow proportional composite.

1.4. Collection Techniques

1.4.1. When filling a sample container that already contains premeasured preservative, slowly pour the sample down the side of the container so that the preservative does not

splatter. If the preservative is concentrated acid, and the sample water is added too quickly, the reaction between the water and the acid can generate enough heat to burn unprotected skin or could splatter and cause acid burning.

1.4.2. Collect grab samples (single, discrete samples) unless directed by permit, program, or approved sampling plan or work plan to collect composite samples.

1.4.3. Except for volatile organic compounds and sulfide, leave ample headspace in the sample bottle to allow for expansion, effervescence and proper mixing at the laboratory.

1.5. Collecting Filtered/Dissolved Samples

1.5.1. Certain studies or projects require collection of dissolved (i.e., filtered) samples. Identify all analytes in samples that are filtered as "dissolved" or "filtered" in field notes or laboratory transmittal forms and on final reports.

1.5.2. Collect both filtered and unfiltered samples from the same water in a collection device (e.g., bailer, intermediate container) or consecutively if sampling from a pump.

1.5.3. Collect dissolved metals in groundwater according to the procedures discussed in FS 2225. **Do not** collect filtered samples for metals from groundwater sources unless:

1.5.3.1. The DEP has required or approved the protocol and the DEP program allows the use of the procedure; or

1.5.3.2. The organization is documenting that a filtered groundwater sample is as or more representative of the groundwater quality. In this case, collect **both** unfiltered and filtered samples for analysis. Submit the results of both samples the DEP for review.

1.5.4. Filtration, when performed, must begin within 15 minutes of sample collection.

1.5.5. Collect dissolved groundwater samples for metals with a one-piece molded construction 1 µm filter unless otherwise specified by a DEP program. Use a 0.45 µm filter when filtering all other constituents **including** metals in surface water.

1.5.6. The filter must be compatible with the analyte to be filtered (e.g., zero carbon content for carbon analysis; non-protein binding filters for nitrogen).

1.5.7. Equipment blanks, when collected, must be processed through the filtration apparatus and analyzed for the analytes of interest.

1.5.8. Filters and filtration equipment are intermediate devices and therefore must be adequately rinsed per FS 2110 section 1.1.2.1.

THE FOLLOWING ARE SPECIAL CONSIDERATIONS FOR VARIOUS ANALYTE GROUPS:

FS 2001. *pH-Preserved Samples*

1. SAMPLE CONTAINERS

1.1. Use properly cleaned sample containers (see FC 1300).

1.2. Inspect all containers for visual defects or contamination. Discard if defects are present or containers do not appear clean.

2. SAMPLE COLLECTION PROCEDURES

2.1. Perform any filtration **before** the sample is poured into the container and **before** the sample is preserved.

2.2. Remove the cap from the sample container, and carefully pour the sample into the container without allowing sampling equipment or hands to touch the rim of the sample container.

2.3. If the preservative is added after the sample is collected, (the container is not prepreserved), do not fill the container to the rim.

3. PRESERVATION

3.1. Preserve the sample within 15 minutes of sample collection or filtration (if applicable) unless collected as a composite sample (see FS 1006, section 1.3) or for analysis of lead and copper for drinking water compliance (see FS 2310, section 2).

3.2. Preserve the sample with the chemical specified by the method or preservation tables (Tables FS 1000-4 to FS 1000-10).

3.2.1. The chemical reagents must be pure enough so that the reagent does not contribute contamination or interferences to the analytes of interest.

3.3. Preserve the sample by adding an accurately measured amount of preservative to the container. Premeasured vials of the preservative, or a graduated container or pipet, may be used.

3.3.1. Tightly cap the sample container and gently tip the container two to three times to distribute the chemical.

3.4. The pH of the preserved sample must meet the pH criterion of the applicable preservation tables (see Tables FS 1000-4 to FS 1000-10). **Do not over preserve the sample.**

3.4.1. Pour an aliquot of the preserved sample into a disposable container (e.g., sampling cup) or onto a piece of **narrow** range pH paper to determine if the pH meets the required level. **Do not put the pH paper directly into the sample container.**

3.4.2. If the pH does not meet the required level, add additional measured amounts of preservative and test with narrow range pH paper (see section 3.4.1 above) until the pH meets the pH requirement.

3.4.3. Record the total amount of preservative that was added to the sample. This documentation is necessary for the next site visit, since additional acid may be needed to adequately preserve the sample on subsequent visits.

3.5. Cooling to less than 6°C with wet ice (see FS 1006, section 5) may be required.

3.6. Protect from direct sunlight.

3.7. Preserve at least one of the equipment blanks with the **greatest** amount of preservative that was required in the sample set and note the amount in field documentation.

3.8. After the sample has been preserved, screw the cap on tightly.

4. Verifying pH-Preserved Samples: Verify the pH of all pH-preserved samples (except volatile organics) in the field (see FS 2001, section 3.4). If samples are routinely collected from the same sample location, a pH check is not required each time samples are collected.

4.1. If the frequency of sample collection at a specified location is once per month or greater (e.g., weekly or daily), check the pH of **at least one** sample per parameter group according to the following schedule:

4.1.1. Weekly sampling: 1 pH check per month

4.1.2. Daily sampling: 1 pH check per week

4.2. If the frequency of sample collection at a specified location is once per month, check the pH of at least one sample per parameter group (except volatile organics) quarterly.

4.2.1. If site conditions vary from sampling event to sampling event, perform pH checks at increased intervals.

4.2.2. For all other sample collection frequencies, pH checks may be reduced as follows:

4.2.2.1. During the first sampling event at a particular site, check **all** samples (except volatile organics) that are pH-adjusted, and

4.2.2.2. During subsequent visits to a particular site, check **at least one** sample per parameter group that must be pH-adjusted.

5. DOCUMENTATION

5.1. Complete the sample container label and stick firmly on the container.

5.2. Complete the field notes.

5.3. Make notes on the transmittal form and in field records about any relevant observations or problems such as entrained sediment or preservation problems.

FS 2002. *Metals*

1. SAMPLE CONTAINERS

1.1. Use properly cleaned containers (see FC 1300).

1.2. Inspect the containers and caps for visual defects or contamination. Do not use containers if defects are present or if they do not appear clean.

2. SAMPLE COLLECTION PROCEDURES

2.1. Perform any filtration **before** the sample is poured into the container and **before** the sample is preserved.

2.2. Remove the cap from the sample container and carefully pour the sample into the container without allowing sampling equipment or hands to touch the rim of the sample container.

3. PRESERVATION - Follow preservation procedures outlined in FS 2001 above.

3.1. Requirements for specific metals:

3.1.1. For boron or cold-vapor atomic absorption Mercury with a grade of nitric acid (HNO_3) that is suitable for use for metals analysis. Use concentrated HNO_3 or 1:1 HNO_3.to lower the pH of less than 2 S.U., but greater than 1.62 S.U.

3.1.2. For Chromium VI add sufficient ammonium sulfate buffer solution specified per Table FS 1000-4 to the sample to raise the pH of the sample to a pH of 9.3 – 9.7 and place in ice (see FS 2002).

3.1.3. Trace Level Mercury

3.1.3.1. Collect samples for trace level mercury (<100 ug/L) in tightly-capped fluoropolymer or glass bottles.

3.1.3.2. If the samples cannot be received by the laboratory within 48 hours of sample collection, preserve the sample with BrCl or HCl solution.

3.1.3.3. For dissolved trace level mercury, samples must be filtered through a 0.45 µm filter within 24 hours of sample collection. If the samples cannot be transported to the laboratory within 24 hours, follow the procedures in FS 8200 for field filtration.

3.1.4. Samples collected for lead and copper for drinking water compliance and metals other than those listed above do not require immediate acid preservation.

3.1.4.1. When samples are not acidified with acid, the transmittal form to the laboratory must:

- Clearly state that the samples are unpreserved; and
- Request that the laboratory preserve the samples.

3.1.4.2. If samples are acidified, use concentrated HNO_3 or 1:1 HNO_3.to lower the pH of less than 2 S.U., but greater than 1.62 S.U.

3.2. After the sample has been preserved, screw the cap on tightly.

4. DOCUMENTATION

4.1. Complete the sample container label and stick firmly on the container.

4.2. Complete the field notes.

4.3. Make notes on the transmittal form and in field records about any relevant observations or problems such as entrained sediment.

4.4. On the transmittal form, clearly identify samples that must be acidified by the laboratory (FS 2002, 3.1.3 or 3.1.4 above).

FS 2003. *Extractable Organics*

1. SAMPLE CONTAINERS

1.1. Most samples are collected in glass containers with Teflon-lined caps. Note: Teflon containers are also acceptable. There are some exceptions such as collecting samples in amber glass (e.g., nitroamines, nitroaromatics, etc.). If in doubt, verify the proper container type in Tables FS 1000-4 through FS 1000-10.

1.2. Inspect glass bottles to assure that there are no visual glass or liner defects. If defects are present and/or the sample containers do not appear clean, the bottles must be discarded.

1.3. Collect composite samples from automatic sample collection devices in refrigerated glass or Teflon containers through Teflon, polyethylene or polypropylene tubing.

2. SAMPLE COLLECTION PROCEDURES

2.1. Remove the cap from the sample container without touching the interior Teflon liner.

2.2. Carefully pour the sample into the container without allowing sampling equipment or hands to touch the rim of the sample container.

2.3. Fill bottle with sample to almost full capacity.

3. PRESERVATION

3.1. In general, these types of samples must be preserved by cooling to 4°C.

3.1.1. Some analyte groups require a chemical preservation. See Tables FS 1000-4 through FS 1000-10 for any additional preservation.

3.1.2. If the samples for pesticides cannot be extracted within 72 hours of collection, the sample pH must be in the range of 5 to 9. If needed, adjust sample to the specified pH range with sodium hydroxide or sulfuric acid.

3.1.3. Add sodium thiosulfate if residual chlorine is present.

3.2. Place samples in **wet** ice within 15 minutes of sample collection (see FS 1006, section 5).

4. DOCUMENTATION

4.1. Complete the sample container label and stick firmly on the container.

4.2. Document when samples were placed in wet ice immediately (see FS 1006, section 5).

4.3. Complete the field notes.

4.4. Make notes on the lab transmittal form and the field records about any sample that appears highly contaminated or exhibits other abnormal characteristics (i.e., foaming, odor, etc.).

FS 2004. *Volatile Organics*

1. SAMPLE CONTAINERS

1.1. Use a screw cap glass sample vial that is sealed with a Teflon-coated septum.

1.2. Collect **at least two** vials of each sample. Some laboratories may require three or more vials, therefore verify the laboratory's policy on the number of vials they require unless the laboratory provides the sampling kit.

1.3. Inspect the vials for glass or septum defects (e.g., rim must not have nicks or visible depressions and the septum must not be deformed). Do not use containers if defects are present or if they do not appear clean.

2. SAMPLE COLLECTION PROCEDURES

2.1. Special precautions for petroleum sources:

2.1.1. If possible, transport and store fuels in a separate vehicle from sampling equipment, empty vials and collected samples. If these items must be transported in the same vehicle as fuel, store the fuels as far away from the vials as possible.

2.1.2. Place all fuel or exhaust sources downwind of the sampling location.

2.1.3. Position all petroleum-fueled engines (including the vehicle) downwind of the sampling operations.

2.2. Do not allow the sampling equipment or hands to touch the rim of the sample container.

2.3. Do not remove septum caps from VOC vials until just prior to filling. Cap vials immediately after filling with sample.

2.4. **DO NOT PRERINSE VOC VIALS.**

2.5. Do not aerate the sample during sample collection. If collecting from a spigot or pump, reduce the flow rate to less than 100 mL/min.

2.6. If preservation is required, proceed to section 3 below unless the laboratory supplied vials with premeasured quantities of acid, and the sample does not need to be dechlorinated (see 3.2 below).

2.6.1. If no preservation is required or if the vials are prepreserved (see 2.5 above), slowly and carefully allow the sample to flow down the **side** of the vial to minimize turbulence. Fill the vial until the surface tension holds the water in a "convex meniscus".

2.6.2. If a vial overflows during the filling process, document the problem and notify the laboratory that the vial may not contain sufficient acid.

2.6.3. If using a bailer, the bailer must be equipped with a controlled flow bottom assembly.

3. PRESERVATION

3.1. Preserve the sample **during** the sample collection process.

3.2. Dechlorination: Some treated water samples (drinking water and treated wastewater) may contain residual chlorine that must be removed with a declorination agent such as sodium thiosulfate or ascorbic acid. This process must occur **before** any additional preservatives (i.e., acid) are added. The dechlorination agent must be **in the vial** before the sample is added.

3.2.1. Laboratories may supply vials with premeasured quantities of declorination agent. If acid preservation **is not required**, fill the vials (see section 2.5.1 above) and proceed to section 4 below.

3.2.2. For chlorinated drinking water samples, add 3 mg sodium thiosulfate per 40 mL vial.

3.2.3. If the chlorine level is unknown, the concentration must be measured (see FT 2000). For sources other than drinking water (e.g., chlorinated effluent), 10 mg sodium thiosulfate per 40 mL vial will remove up to 5 ppm Cl_2.

3.3. Acid Preservation

3.3.1. Chlorinated Samples

3.3.1.1. If acid preservation is required, carefully fill the vial with sample, but not to a convex meniscus as described in section 2.5.1 above.

3.3.1.2. Add four drops of concentrated HCl (more acid may be needed if the sample is known to contain high levels of bicarbonate or is otherwise buffered).

3.3.1.3. Add additional sample to create a convex meniscus.

NOTE: If the sample reacts with the acid by generating gas, do not submit preserved samples for analysis. Instead, collect unpreserved samples (seven-day holding time must be met).

3.3.2. Unchlorinated Samples

3.3.2.1. The laboratory may supply vials with premeasured quantities of acid. In this case, proceed to section 2.5.1 above. If a vial overflows during the filling process, document the problem and notify the laboratory that the vial may not contain sufficient acid.

3.3.2.2. If the samples are preserved in the field, follow the procedure in section 3.3 above.

4. CAPPING THE VIAL

4.1. Fill the vial so that the sample surface is above the container rim (convex meniscus).

4.1.1. **Do not pour** sample into cap.

4.1.2. Fill vial from the original source (tubing, spigot, etc.) **Do not fill vial from sample collected in the cap**.

4.2. **Immediately** cap the vial with the Teflon seal contacting the sample. Some sample may overflow while tightening the cap.

4.3. If acid has been added to the sample, tip the vial gently two or three times to distribute the preservative.

4.4. Turn the vial over and tap it to check for the presence of bubbles.

4.4.1. If bubbles are present, and the total volume of the bubbles is less than 5 mm in diameter, the sample may be submitted.

4.4.2. If the total volume of the bubbles is greater than 5 mm in diameter, discard the vial and fill a new one.

4.4.3. **Do not open a vial to add additional sample.**

5. SAMPLE PACKING

5.1. Label each vial with an appropriate field ID number and preservation (e.g., preserved with acid, sodium thiosulfate/acid, etc.).

5.2. Wrap each vial in a protective material (e.g., bubble wrap).

5.3. Place the set of vials in a small, sealable, untreated plastic bag unless the laboratory supplies an alternate method of packing.

5.4. Place samples in **wet** ice within 15 minutes of sample collection (see FS 1006, section 5).

5.5. Protect samples from environmental contamination during storage and transport to the laboratory.

5.6. As an added measure, DEP recommends wrapping the set of replicate samples in bubble wrap and sealing them in a container. This procedure will add further protection from potential contamination.

6. DOCUMENTATION

6.1. Label all the vials.

6.2. Complete field records.

6.3. Make note in the field records of any samples that appear highly contaminated or appear to effervesce when acid is added.

FS 2005. *Bacteriological Sampling*

1. SAMPLE CONTAINERS

1.1. Collect the samples in properly sterilized containers.

1.1.1. Presterilized Whirl-pak bags (or equivalent) are generally used.

1.1.2. If Whirl-pak bags are not used, the sample container must have a volume of at least 125 mL.

1.1.3. If using bottles, the caps must be sterilized. If the caps are lined, there must be documentation to show that the liner does not produce toxic compounds when sterilized.

1.1.4. Bottles and caps must be sterilized according to procedures in FC 1320 or purchased presterilized from a commercial vendor.

2. SAMPLE COLLECTION PROCEDURES

2.1. Unless a composite is specified by permit, all samples must be grab samples.

2.2. Do not open the container once it has been sealed.

2.3. Do not rinse sample container before collecting the sample.

2.4. Use aseptic techniques to collect the sample:

2.4.1. If an intermediate device is used, thoroughly rinse with sample water. To ensure proper rinsing, DEP recommends that microbiological samples be the last sample collected with the sampling device.

2.4.2. Do not put fingers into the mouth of the container or on the interior of the cap.

2.4.3. Do not disinfect the sample equipment or sampling port.

2.5. Rinse the sampling equipment with sample water before collecting the sample. Therefore, collect microbiological samples at the end of a sampling sequence.

2.6. Wells with In-Place Plumbing, Spigots and/or Faucets

2.6.1. Do not disinfect the spigot with bleach, alcohol or heat. Turn on spigot and flush at maximum velocity (see FS 2310).

2.6.2. After flushing, reduce the water flow to approximately 500 mL/min and allow the water to flow for a few minutes before collecting samples. If other samples (metals, nutrients, etc.) are to be collected, collect these samples first.

2.6.3. **Do not stop the flow before or during the filling process.**

2.7. Direct Grab Sample Collection

2.7.1. Hold a rigid container near the base and plunge neck downward, below the surface. Turn container until the neck points slightly upward with the mouth directed toward the current. Fill to within about 1/2 inch of the top and cap immediately.

2.7.2. Whirl-pak bags (or equivalent)

- Open the bag by zipping off the top and pulling the white tabs to open the bag. Hold the bag behind the wire ties, and plunge neck downward and up in one sweeping arc; or
- Zip off the top of the bag. Hold bag so that the mouth and wire ties are in front of the hands and fingers. Immerse the bag, and open the bag into the current.
- The above procedures may also be accomplished by attaching the bag to a pole.

2.7.2.1. Bring the bag to the surface, and press out excess water.

2.7.2.2. Seal the bag by folding the open ends at least three times and securely twisting the wire ties.

2.8. Intermediate Device Collection

2.8.1. When using an intermediate sampling device (bailer, DO dunker, niskin bottle, etc.), obtain sufficient sample in the sample collection device to completely fill the sample container. Begin pouring sample out of the device BEFORE collecting into the container. Continue to pour sample out of the device, place container under flowing stream, and fill. **Do not stop the flow before or during the filling process.**

3. PRESERVATION

3.1. Preserve samples according to Tables FS 1000-4 through FS 1000-10.

3.2. Place all samples in wet ice immediately after sample collection (see FS 1006, section 5).

3.3. When the sample contains residual chlorine, add a dechlorinating agent such as sodium thiosulfate to the sample container.

3.3.1. The final concentration of sodium thiosulfate must be approximately 100 milligrams per liter (mg/L) in the sample (add 0.1 mL of a 10% solution of thiosulfate to a 125 mL sample).

3.3.2. Some vendors or laboratories provide sterile containers with premeasured amounts of dechlorinating agent. Determine if the source of the field containers already contain a dechlorinating agent.

3.3.3. **Do not use containers with dechlorinating chemicals** when collecting samples from sources that are known to be free from residual chlorine.

4. HOLDING TIME

4.1. The holding time for microbiological samples is very short. Let the laboratory know the approximate time that samples will be collected and when they are expected to be delivered to the laboratory.

4.2. The holding time begins at the time (hours and minutes) the sample is collected and ends at the time that the sample is placed on the applicable growth media.

4.3. Consult Tables FS 1000-4, -6, -8, and -9 for holding times.

5. DOCUMENTATION

5.1. Label each sample container with an appropriate field ID number.

5.2. Place samples in **wet** ice within 15 minutes of sample collection (see FS 1006, section 5).

5.3. Complete field records.

5.4. Make note in the field records of any unusual sample appearances or sampling conditions.

FS 2006. *Oil and Grease (O&G) and Total Recoverable Petroleum Hydrocarbons (TRPHs)*

1. SAMPLE CONTAINERS

1.1. Collect samples for O&G and TRPHs in 1-liter wide mouth amber glass bottles.

1.2. The cap must have a Teflon liner.

1.3. Visually inspect glass bottles and caps for defects. Do not use container if defects are present or if they do not appear clean.

2. SELECTION OF SAMPLING POINTS

2.1. Oil and grease may be present in wastewater as a surface film, an emulsion, a solution, or as a combination of these forms. Since it is very difficult to collect a representative ambient sample for oil and grease analysis, the sampler must carefully evaluate the location of the sampling point.

2.1.1. Select a point of greatest mixing.

2.1.2. For compliance samples at a facility, collect samples from a point that best represents oil and grease concentrations.

3. SAMPLE COLLECTION PROCEDURES

3.1. All samples must be grab samples.

3.1.1. If composite data are required, collect individual grab samples over the specified time period.

3.1.2. Submit all samples for analysis.

3.1.3. Average the concentrations of the results to determine the average concentration over time.

3.2. Do not collect the sample by skimming the surface.

3.3. Collect a discrete sample that will be used for analysis. Do not use this sample for any other test.

3.4. Remove the cap from the glass bottle without touching the interior of the container or lid.

3.5. Do not rinse the sampling device or the sample container with sample water.

3.6. Collect the sample directly into the container.

3.6.1. If intermediate sampling equipment is needed, do not allow the sampling equipment to touch the rim of the sample container.

3.6.2. Do not use automatic samplers to collect these types of samples.

3.6.3. Fill the bottle with the sample water to almost full capacity.

3.6.4. Add preservatives (see section 4 below).

3.6.5. Quickly cap the container and tighten securely.

4. PRESERVATION

4.1. Preserve the sample within 15 minutes of sample collection.

4.2. The pH of the acidified sample must be less than 2. **Do not over acidify the sample.**

4.3. Preserve the sample by adding an accurately measured amount of sulfuric or hydrochloric acid to the container. Premeasured vials of acid, or a graduated container or pipet, may be used.

4.3.1. Tightly cap the sample container and shake to distribute the acid.

4.3.2. Pour an aliquot of the acidified sample into a disposable container (e.g., sampling cup) or onto a piece of **narrow** range pH paper to determine if the pH is less than 2. **Do not put the pH paper directly into the sample container.**

4.3.3. If the pH is greater than 2, add additional measured amounts of acid and test with narrow range pH paper (see section 4.3.2 above) until the pH has been reduced to below 2 pH units.

4.3.4. Record the total amount of acid that was added to the sample.

4.4. Acidify at least one of the equipment blanks with the **greatest** amount of acid that was required in the sample set and note the amount in field documentation.

4.5. After the sample has been preserved, screw the cap on tightly.

4.6. Immediately place the sample in **wet** ice after preserving with acid (see FS 1006, section 5).

5. DOCUMENTATION

5.1. Label each vial with an appropriate field ID number.

5.2. Protect glass container from breakage ("bubble wrap" is recommended).

5.3. Complete field records.

5.4. Make notes on the transmittal form and in field records about any relevant observations or problems such as entrained sediment.

FS 2007. *Radiological Sampling (Excludes Radon)*

1. SAMPLE CONTAINERS

1.1. Use polyethylene, polyvinyl chloride (PVC), or Teflon containers.

1.2. Visually inspect the containers and caps for defects. If defects are present and/or sample containers do not appear to be clean, do not use the containers.

2. SAMPLE COLLECTION PROCEDURES

2.1. On unknown sites, survey the area with a beta-gamma survey instrument, such as a Geiger-Müller meter.

2.1.1. If radiation levels are above instrument background, consult a radiation safety specialist to determine appropriate safety procedures.

2.2. Remove the cap from the sample container and carefully pour the sample into the container without allowing sampling equipment or hands to touch the rim of the sample container.

3. PRESERVATION

3.1. Preserve the sample with a suitable grade of nitric acid (HNO_3).

3.2. Preserve the sample within 15 minutes of sample collection.

3.3. The pH of the acidified sample must be less than 2. **Do not over acidify the sample.**

3.4. If the preservative is added after the sample is collected (the container is not prepreserved), do not fill the container to the rim.

3.5. Preserve the sample by adding an accurately measured volume of concentrated HNO_3 or 1:1 HNO_3 to the container. Premeasured vials of acid, or a graduated container or pipet, may be used.

3.5.1. Tightly cap the sample container and shake to distribute the acid.

3.5.2. Pour an aliquot of the acidified sample into a disposable container (e.g., sampling cup) or onto a piece of **narrow** range pH paper to determine if the pH is less than 2. **Do not put the pH paper directly into the sample container.**

3.5.3. If the pH is greater than 2, add additional measured amounts of acid and test with narrow range pH paper (see section 3.5.2 above) until the pH has been reduced to just below 2 pH units.

3.5.4. Record the total amount of acid that was added to the sample.

3.5.5. Cooling to 4°C is not required.

3.6. Acidify at least one of the equipment blanks with the **greatest** amount of acid that was required in the sample set and note the amount in field documentation.

3.7. After the sample has been preserved, screw the cap on tightly.

4. DOCUMENTATION

4.1. Complete the sample container label and stick firmly on the container.

4.2. Complete the field notes.

4.3. Make notes on the transmittal form and in field records about any relevant observations or problems such as entrained sediment.

FS 2008. *Radon Sampling*

Radon is a gas and is easily removed from water sources. Therefore, follow the same precautions and care used to collect volatile organic samples. Minimize contact with air during sample collection. Other sample collection techniques may be appropriate, depending on the analytical method or as specified in the project data quality objectives.

1. SAMPLE CONTAINERS

1.1. Use glass sample vials containing a premeasured portion of the scintillation "cocktail."

1.2. Visually inspect the containers and caps for defects. If defects are present and/or sample containers do not appear to be clean, do not use the containers.

1.3. Collect at least two samples.

2. PRESERVATION: The scintillation cocktail is the only required preservative.

3. SAMPLE COLLECTION PROCEDURES Obtain specific sample collection instructions from the laboratory that will analyze the samples. These instructions must include proper handling as well as sample size and packing instructions. The following are general instructions for collecting the samples:

3.1. Carefully fill a syringe (usually 10 mL) with sample water so that air bubbles are not pulled in with the sample before, during or after filling.

3.2. Place the tip of the syringe BELOW the scintillation cocktail and slowly dispense the sample BENEATH the cocktail surface.

3.3. Replace the lid and cap tightly.

3.4. Generally, the vial is used in the laboratory analytical instrument and labels or ID numbers on the sides of the containers may interfere with the analysis. Check with the laboratory for proper placement of labels or field ID numbers.

3.5. Ship in an upright position in the shipping containers that have been provided by the laboratory. If none are provided, protect vials from breakage ("bubble wrap" is recommended), segregate replicate samples in separate plastic bags, and ship to the laboratory in an upright position.

4. DOCUMENTATION

4.1. Complete the field notes.

4.2. Make notes on the transmittal form and in field records about any relevant observations or problems such as entrained sediment.

FS 2009. *Cyanide Sampling*

Cyanide is a very reactive and unstable species and is highly toxic. Samples suspected of containing cyanide must be handled very carefully.

1. SAMPLE CONTAINERS

1.1. Use polyethylene or glass sample containers.

1.2. Use properly cleaned containers (see FC 1300).

1.3. Visually inspect the containers and caps for defects. If defects are present and/or sample containers do not appear to be clean, do not use the containers.

2. SAMPLE COLLECTION PROCEDURES

2.1. Remove the cap from the sample container, and carefully pour the sample into the container without allowing sampling equipment or hands to touch the rim of the sample container.

3. PRESERVATION

3.1. Many different analytes interfere with the cyanide analysis (e.g., sulfides). If any interferences are known to be present, pretreat the sample for interferences by following the applicable footnotes in Table FS 1000-4.

3.2. Preserve the sample within 15 minutes of sample collection.

3.3. Preserve samples with sodium hydroxide to a pH greater than 12.

3.4. Preserve the sample by adding an accurately measured amount of a sodium hydroxide solution or sodium hydroxide pellets to the container. Use a graduated container or pipet to add the solution.

3.4.1. Tightly cap the sample container and shake to distribute the preservative.

3.4.2. Pour an aliquot of the preserved sample into a disposable container (e.g., sampling cup) or onto a piece of **narrow** range pH paper to determine if the pH is greater than 12. **Do not put the pH paper directly into the sample container.**

3.4.3. If the pH is less than 12, add additional measured amounts of the preservative and test with narrow range pH paper (see section 3.4.2 above) until the pH has been raised to above 12 pH units.

3.4.4. Record the total amount of preservative that was added to the sample.

3.5. After the sample has been preserved, screw the cap on tightly.

3.6. Immediately put the sample in **wet** ice (see FS 1006, section 5).

3.7. Preserve at least one of the equipment blanks with all the reagents and the **greatest** amount of sodium hydroxide that was required in the sample set and note the amount in field documentation.

4. DOCUMENTATION

4.1. Complete the sample container label and stick firmly on the container.

4.2. Complete the field notes.

4.3. Make notes on the transmittal form and in field records about any relevant observations or problems such as entrained sediment.

4.4. Ensure that all preservation measures are part of the field notes.

FS 2010 *Sulfide Sampling*

1. Analyze samples within 15 minutes of collection, or the preserve the sample within 15 minutes for later analysis. If preservation is required add the zinc acetate and sodium hydroxide to the container **before** filling with sample.

2. Avoid aerating the sample during collection. Slowly pour the sample slowly and carefully allow the sample to flow down the **side** of the container to minimize turbulence.

3. Check the pH (if necessary) before completing the filling process.

4. Complete the filling process. **Do not leave a head space.**

FS 2200. Groundwater Sampling

1. INTRODUCTION AND SCOPE

1.1 Use these Standard Operating Procedures to collect groundwater samples. They are designed to ensure that the collected samples will be representative of water in the aquifer or target formation and that the samples have not been altered or contaminated by the sampling and handling procedures. These procedures apply to permanently and temporarily installed monitoring wells, wells constructed using "direct-push" techniques, wells with installed plumbing, remedial groundwater treatment systems and excavations where groundwater is present. Use of alternative, DEP-approved and properly documented procedures (e.g., Corporate SOP, ASTM Standards, alternative equipment, etc.) is acceptable if they meet the intent (e.g., sample representativeness and integrity) of this standard (see FA 1000).

1.2 The topics in this SOP include equipment and supply selection, equipment construction materials, and purging and sampling techniques.

1.3 Use the following DEP SOPs in conjunction with FS 2200:

- FA 1000 Regulatory Scope and Administrative Procedures for Use of DEP SOPs
- FC 1000 Cleaning/Decontamination Procedures
- FD 1000 Documentation Procedures
- FQ 1000 Field Quality Control Requirements
- FS 1000 General Sampling Procedures
- FS 2000 General Aqueous Sampling
- FT 1000 Field Testing and Measurement
- FT 1100 Field pH
- FT 1200 Field Specific Conductance
- FT 1400 Field Temperature
- FT 1500 Field Dissolved Oxygen
- FT 1600 Field Turbidity

2. Groundwater samples may be collected from a number of different configurations. Each configuration is associated with a unique set of sampling equipment requirements and techniques:

3. Wells without Plumbing: These wells require that equipment be brought to the well to purge and sample unless dedicated equipment is placed in the well.

4. Wells with In-Place Plumbing: Wells with in-place plumbing do not require that equipment be brought to the well to purge and sample. In-place plumbing is generally considered permanent equipment routinely used for purposes other than purging and sampling, such as for water supply. They are generally found at wellfields, industrial facilities, and private residences. See FS 2300 for procedures to sample potable water wells. Air Strippers or Remedial Systems: These types of systems are installed as remediation devices. Sample these wells like drinking water wells (see FS 2300).

FS 2201 *Equipment and Supplies*

Use groundwater purging and sampling equipment constructed of only non-reactive, non-leachable materials that are compatible with the environment and the selected analytes. In selecting groundwater purging and sampling equipment, give consideration to the depth of the well, the depth to groundwater, the volume of water to be evacuated, the sampling and purging technique, and the analytes of interest. Refer to Tables FS 1000-1, FS 1000-2, FS 1000-3 and FS 2200-1 for selection of appropriate equipment.

Additional supplies such as reagents, preservatives, and field measurement equipment are often necessary.

1. FLOW CONTAINER: DEP recommends using a flow-through cell or container when collecting measurements for purging stabilization. The design must ensure that fresh formation water continuously contacts the measuring devices and does not aerate the sample or otherwise affect the groundwater properties.

2. PUMPS: All pumps or pump tubing must be lowered and retrieved from the well slowly and carefully to minimize disturbance to the formation water. This is especially critical at the air/water interface. Avoid the resuspension of sediment particles (turbidity) at the bottom of the well or adhered to the well casing during positioning of the pump or tubing.

2.1 Above-Ground Pumps

2.1.1 Variable Speed Peristaltic Pump: Use a variable speed peristaltic pump to purge groundwater from wells when the static water level in the well is no greater than 20-25 feet below land surface (BLS). If the water levels are deeper than 18-20 feet BLS, the pumping velocity will decrease.

2.1.1.1 A variable speed peristaltic pump can be used for normal purging and sampling (see FS 2213 and FS 2221), sampling low permeability aquifers or formations (see FS 2222) and collecting filtered groundwater samples (see FS 2225, section 1).

2.1.1.2 Most analyte groups can be sampled with a peristaltic pump if the tubing and pump configurations are appropriate. See Table FS 1000-3 for proper tubing selection and pump configurations.

2.1.2 Variable Speed Centrifugal Pump: A variable speed centrifugal pump can be used to purge groundwater from 2-inch and larger internal diameter wells. Do not use this type of pump to collect groundwater samples.

2.1.2.1 When purging is complete, do not allow the water that remains in the tubing to fall back into the well. Install a check valve at the end of the purge tubing, and withdraw the tubing slowly from the well while the pump is still running.

2.1.2.2 See Table FS 1000-3 for proper tubing selection and allowable analyte groups.

2.2 Submersible Pumps

2.2.1 Variable Speed Electric Submersible Pump: A variable speed submersible pump can be used to purge and sample groundwater from 2-inch and larger internal diameter wells.

2.2.1.1 A variable speed submersible pump can be used for normal purging and sampling (see FS 2213 and FS 2221), sampling low permeability aquifers or

formations (see FS 2222) and collecting filtered groundwater samples (see FS 2225, section 1).

2.2.1.2 Make sure that the pump housing, fittings, check valves and associated hardware are constructed of stainless steel. Make sure that any other materials are compatible with the analytes of interest. See Table FS 1000-3 for restrictions.

2.2.1.3 Install a check valve at the output side of the pump to prevent backflow.

2.2.1.4 If purging and sampling for organics:

- The entire length of the delivery tube must be Teflon, Polyethylene or Polypropylene (PP) tubing.
- The electrical cord must be sealed in Teflon, Polyethylene or PP and any cabling must be sealed in Teflon, Polyethylene or PP, or be constructed of stainless steel.
- All interior components that contact the sample water (impeller, seals, gaskets, etc.) must be constructed of stainless steel or Teflon.

2.2.2 Variable Speed Bladder Pump: A variable speed positive displacement bladder pump (no-gas contact) can be used to purge and sample groundwater from 3/4-inch and larger internal diameter wells.

2.2.2.1 A variable speed bladder pump can be used for normal purging and sampling (see FS 2213 and FS 2221), sampling low permeability aquifers or formations (see FS 2222) and collecting filtered groundwater samples (see FS 2225, section 1).

2.2.2.2 The bladder pump system is composed of the pump, the compressed air tubing, the water discharge tubing, the controller and a compressor or compressed gas supply.

2.2.2.3 The pump consists of a bladder and an exterior casing or pump body that surrounds the bladder and two (2) check valves. These parts can be composed of various materials, usually combinations of polyvinyl chloride (PVC), Teflon, Polyethylene, PP and stainless steel. Other materials must be compatible with the analytes of interest. See Table FS 1000-3 for restrictions.

2.2.2.4 If purging and sampling for organics:

- The pump body must be constructed of stainless steel and the valves and bladder must be Teflon, Polyethylene or PP̃
- The entire length of the delivery tube must be Teflon, Polyethylene or PP.
- Any cabling must be sealed in Teflon̩ Polyethylene or PP, or be constructed of stainless steel.
- Permanently installed pumps may have a PVC pump body as long as the pump remains in contact with the water in the well.

3. BAILERS:

3.1 Purging: DEP does not recommend using bailers for purging unless no other equipment can be used or purging with a bailer has been specifically authorized by a DEP program, permit, contract or order (see Table FS 2200-3). Use a bailer if there is non-aqueous phase liquid (free product) in the well or non-aqueous phase liquid is suspected to

be in the well. If in doubt about the appropriateness of using a bailer at a site or during a particular sampling event, contact the appropriate DEP program or project manager. If a bailer is used, follow FS 2213, section 4, with no deviations.

3.2 Sampling: Bailers may be used to routinely collect some analyte groups or under specific circumstances for other analyte groups (see Table FS 2200-3).

3.3 Construction and Type:

3.3.1 Bailers must be constructed of materials compatible with the analytes of interest. See Table FS 1000-3 for restrictions.

3.3.2 Stainless steel, Teflon, Polyethylene and PP bailers may be used to sample all analytes.

3.3.3 Use disposable bailers when sampling grossly contaminated sample sources.

3.3.4 DEP recommends using dual check valve bailers when collecting samples.

3.3.5 Use bailers with a controlled flow bottom when collecting volatile organic samples.

3.3.6 Use bailers that can be pressurized when collecting filtered samples for metals.

3.4 Contamination Prevention:

3.4.1 Keep the bailer wrapped (foil, butcher paper, etc.) until just before use.

3.4.2 Use protective gloves to handle the bailer once it is removed from its wrapping.

3.4.3 Handle the bailer by the lanyard to minimize contact with the bailer surface.

4. LANYARDS

4.1 Lanyards must be made of non-reactive, non-leachable material such as cotton twine, nylon, or stainless steel; or, coated with Teflon, Polyethylene or PP.

4.1.1 Evaluate the appropriateness of the lanyard material with analyses of equipment blanks for the analytes of interest, as necessary.

4.2 Discard cotton twine, nylon, and non-stainless steel braided lanyards after sampling each monitoring well.

4.3 Decontaminate stainless steel, coated Teflon, Polyethylene and PP lanyards between monitoring wells (see FC 1003). They do not need to be decontaminated between purging and sampling operations.

4.4 Securely fasten lanyards to downhole equipment (bailers, pumps, etc.).

4.5 Do not allow lanyards used for downhole equipment to touch the ground surface.

FS 2210. GROUNDWATER PURGING

Perform procedures in the following sections to calculate purging parameters and to purge groundwater from monitoring wells, wells with installed plumbing, high-volume wells, air stripper systems and other remedial treatment systems.

FS 2211 *Water Level and Purge Volume Determination*

Collect representative groundwater samples from the aquifer. The amount of water that must be purged from a well is determined by the volume of water and/or field parameter stabilization.

1. GENERAL EQUIPMENT CONSIDERATIONS

1.1 Selection of appropriate purging equipment depends on the analytes of interest, the well diameter, transmissivity of the aquifer, the depth to groundwater and other site conditions.

1.2 Use a pump to purge the well.

1.3 Use a bailer if there is non-aqueous phase liquid in the well or non-aqueous phase liquid is suspected to be in the well.

1.4 Bailers may be used if approved by a DEP program, or if bailer use is specified in a permit, contract or DEP order (see Table FS 2200-3). If used, bailers must be of appropriate type and construction, and the user must follow the procedure outlined in FS 2213, section 4, with no deviations. If in doubt about the appropriateness of using a bailer at a site or during a particular sampling event, contact the appropriate DEP program or project manager. DEP does not recommend using bailers because improper bailing:

1.4.1 Introduces atmospheric oxygen which precipitates metals (i.e., iron) or causes other changes in the chemistry of the water in the sample (i.e., pH)

1.4.2 Agitates groundwater which biases volatile and semi-volatile organic analyses due to volatilization

1.4.3 Agitates the water in the aquifer and resuspends fine particulate matter

1.4.4 Surges the well, loosening particulate matter in the annular space around the well screen

1.4.5 Introduces dirt into the water column if the sides of the casing wall are scraped

2. INITIAL INSPECTION

2.1 Verify the identification of the monitoring well by examining markings, sign plates, placards or other designations.

2.2 Remove the well cover and remove all standing water around the top of the well casing (manhole) before opening the well cap.

2.3 Inspect the exterior protective casing of the monitoring well for damage and document the results of the inspection if there is a problem.

2.4 It is recommended that you place a protective covering around the well head. Replace the covering if it becomes soiled or ripped.

2.5 Inspect the well lock and determine whether the cap fits tightly. Replace the cap if necessary.

3. WATER LEVEL MEASUREMENTS: Use an electronic probe or chalked tape to determine the water level.

3.1 General Procedures

Perform these steps using either the electronic probe or chalked tape method.

3.1.1 Decontaminate all equipment that will contact the groundwater in the well before use.

3.1.2 Measure the depth to groundwater from the top of well casing to the nearest 0.01 foot and always measure from the same reference point or survey mark on the well casing. If there is no reference mark, measure from the north side of the casing.

3.1.3 Record the measurement and the reference point.

3.2 Electronic Probe

3.2.1 Follow the manufacturer's instructions for use.

3.2.2 Record the measurement.

3.3 Chalked Line Method: This method is not recommended if collecting samples for organic or inorganic parameters.

3.3.1 Lower chalked tape into the well until the lower end is in the water (usually determined by the sound of the weight hitting the water).

3.3.2 Record the length of the tape relative to the reference point (see section 3.2 above).

3.3.3 Quickly remove the tape from the well.

3.3.4 Record the length of the wetted portion to the nearest 0.01 foot.

3.3.5 Determine the depth to water by subtracting the length of the wetted portion (see section 3.5.3 above) from the total length (see section 3.5.2 above). Record the result.

4. WATER COLUMN DETERMINATION

4.1 Do not determine the total depth of the well by lowering the probe to the bottom of the well immediately before purging and sampling. If the well must be sounded, delay purging and sampling activities for at least 24 hours after the well was sounded or for a time sufficient to meet the purge stabilization criterion for turbidity. Alternatively, collect samples before sounding the well.

4.2 Subtract the depth to the top of the water column from the total well depth to determine the length of the water column.

4.3 The total well depth depends on the well construction. Some wells may be drilled in areas of sinkhole or karst formations or rock leaving an open borehole. Attempt to find the total borehole depth in cases where there is an open borehole below the cased portion.

5. WELL WATER VOLUME

5.1 Calculate the total volume of water in gallons in the well using the following equation:

V = (0.041)d x d x h

Where: V = volume in gallons

d = well diameter in inches

h = height of the water column in feet

5.2 The total volume of water in the well may also be determined with the following equation by using a casing volume per foot factor (Gallons per Foot of Water) for the appropriate diameter well:

V = [Gallons per Foot of Water] x h

Where: V = volume in gallons

h = height of the water column in feet

Casing Internal Diameter	Approximate Gallons per Foot of Water
0.75”	0.02
1”	0.04
1.25”	0.06
2”	0.16
3”	0.37
4”	0.65
5”	1.02
6”	1.47
12”	5.88

5.3 Record all measurements and calculations in the field records.

6. Purging Equipment Volume

Calculate the total volume of the pump, associated tubing and container that is used for in situ measurements (flow container), if used, using the following equation:

V = p + ((0.041)d x d x l) + fc

Where: V = volume in gallons

p = volume of pump in gallons

d = tubing diameter in inches

l = length of tubing in feet

fc = volume of flow cell in gallons

7. When collecting samples from multiple wells on a site, if the groundwater elevation data are to be used to construct groundwater elevation contour maps, all water level measurements must be taken within the same 24-hour time interval unless a shorter time period is required by a DEP program. If the site is tidally influenced, complete the water level measurements within the time frame of an incoming or outgoing tide.

FS 2212 *Well Purging Techniques*

The selection of the purging technique and equipment is dependent on the hydrogeologic properties of the aquifer, especially depth to groundwater and hydraulic conductivity. The intent of proper purging is to stabilize the water level in the well and minimize the hydraulic stress to the hydrogeologic formation.

Every attempt must be made to match the pumping rate with the recharge rate of the well before evaluating the purging completion criteria.

A flowchart which summarizes purging procedure options is presented in Figure FS 2200-2.

Select equipment using the construction and configuration requirements specified in Table FS 2200-1. See the discussions in FS 2201.

1. MEASURING THE PURGE VOLUME: The volume of water that is removed during purging must be recorded. Measure the volume during the purging operation.

1.1 Collect the water in a graduated container and multiply the number of times the container was emptied by the volume of the container, or

1.2 Estimate the volume based on pumping rate. Use this technique only if the pumping rate is constant. Determine the pumping rate by measuring the amount of water that is pumped for a fixed period of time or use a flow meter.

1.2.1 Calculate the amount of water that is discharged per minute:

$$D = \frac{\text{Measured amount}}{\text{Total time in minutes}}$$

1.2.2 Calculate the time needed to purge one (1) well volume or one (1) purging equipment volume:

$$\text{Time} = \frac{V}{D}$$

Where: V = well volume determined from FS 2211, section 5, or purging equipment volume

D = discharge rate calculated in section 1.2.1. above

1.2.3 Make new measurements (see section 1.2.1 above) each time the pumping rate is changed, or

1.3 Use a totalizing flow meter.

1.3.1 Record the reading on the totalizer prior to purging.

1.3.2 Record the reading on the totalizer at the end of purging.

1.3.3 Subtract the reading on the totalizer prior to purging from the reading on the totalizer at the end of purging to obtain the volume purged.

1.4 Record in the field records the times that purging begins and ends.

2. Stabilization Measurement Frequency

2.1 Begin to record stabilization measurements after pumping the minimum volume as prescribed in options 2.3 – 2.5 below. Every attempt must be made to match the pumping rate with the recharge rate of the well before evaluating the purging criteria.

2.2 If the well screened interval is not known, use option 2.3, below.

2.3 Wells with Fully Submerged Screen and Pump or Intake Tubing Placed at the Top of the Water Column (conventional purge): Purge until the water level has stabilized (well recovery rate equals the purge rate), then purge a minimum of one (1) well volume prior to collecting measurements of the stabilization parameters. Allow at least one quarter (1/4) well volume to purge between subsequent measurements.

2.4 Wells with Fully Submerged Screen and Pump or Intake Tubing Placed Within the Screened Interval (minimizing purge volume): Purge until the water level has stabilized (well recovery rate equals the purge rate), then purge a minimum of one (1) volume of the pump, associated tubing and flow container (if used) prior to collecting measurements of the stabilization parameters. Take measurements of the stabilization parameters no sooner

than two (2) minutes apart. Purge at least three (3) volumes of the pump, associated tubing and flow container, if used, prior to collecting a sample.

If the water level drops into the screened interval during purging, lower the pump or tubing intake as in FS 2213, section 1.3 below and follow purging procedures for partially submerged well screens (2.5 below).

2.5 Wells with a Partially Submerged Well Screen: Purge until the water level has stabilized (well recovery rate equals the purge rate), then purge a minimum of one (1) well volume prior to collecting measurements of the stabilization parameters. Take measurements of the stabilization parameters no sooner than two (2) minutes apart.

3. PURGING COMPLETION: DEP recommends the use of a flow-through container to measure the stabilization parameters discussed below. Alternatively, measure all parameters *in situ* by inserting measurement probes into the well at the depth appropriate for the purging option. Purging is considered complete if the criteria in section 3.1, 3.2 or 3.3 below are satisfied. Make every attempt to satisfy the criteria in section 3.1. Every attempt must be made to match the pumping rate with the recharge rate of the well before evaluating the purging criteria.

3.1 Three (3) consecutive measurements of the five (5) parameters listed below must be within the stated limits. The measurements evaluated must be the last three consecutive measurements taken before purging is stopped. The range between the highest and the lowest values for the last three measurements of temperature, pH and specific conductance cannot exceed the stated limits. The last three consecutive measurements of dissolved oxygen and turbidity must all be at or below the listed thresholds.

- Temperature: ± 0.2° C
- pH: ± 0.2 Standard Units
- Specific Conductance: ± 5.0% of reading
- Dissolved Oxygen: ≤20% Saturation
- Turbidity: ≤20 NTU

3.2 Naturally occurring conditions may prevent attaining the ≤20% saturation criterion for dissolved oxygen, typically in surficial aquifers. See section 3.5, below.

3.3 Naturally occurring conditions may prevent attaining the ≤20 NTU criterion for turbidity. However, when collecting groundwater samples for metals or certain inorganic (e.g., phosphorus forms) or extractable organic (e.g. polynuclear aromatic hydrocarbons) chemicals, make every attempt to reduce turbidity to ≤20 NTU to avoid a potential turbidity-associated bias for these analytes. See section 3.5, below.

3.4 Document and report the following, as applicable, except that the last four (4) items only need to be submitted once:

- Purging rate.
- Drawdown in the well, if any.
- Pump or tubing intake placement.
- Length and location of the screened interval.
- A description of the process and the data used to design the well.
- The equipment and procedure used to install the well.

- The well development procedure.
- Pertinent lithologic or hydrogeologic information.

3.5 If the criteria in section 3.1 above for dissolved oxygen and/or turbidity cannot be met, then three (3) consecutive measurements of the five (5) parameters listed below must be within the stated limits.

3.5.1 The measurements evaluated must be the last three consecutive measurements taken before purging is stopped. The range between the highest and the lowest values for the last three measurements cannot exceed the stated limits.

- Temperature: ± 0.2° C
- pH: ± 0.2 Standard Units
- Specific Conductance: ± 5.0% of reading
- Dissolved Oxygen: ± 0.2 mg/L or 10%, whichever is greater
- Turbidity: ± 5 NTUs or 10%, whichever is greater

3.5.2 Additionally, document and report the following, as applicable, except that the last four (4) items only need to be submitted once:

- Purging rate.
- Drawdown in the well, if any.
- Pump or tubing intake placement.
- Length and location of the screened interval.
- A description of conditions at the site that cause the dissolved oxygen to be high and/or dissolved oxygen measurements made within the screened or open borehole portion of the well with a downhole dissolved oxygen probe.
- A description of conditions at the site that cause the turbidity to be high and any procedures that will be used to minimize turbidity in the future.
- A description of the process and the data used to design the well.
- The equipment and procedure used to install the well.
- The well development procedure.
- Pertinent lithologic or hydrogeologic information.

3.5.3 If from review of the submitted data the Department determines that both the elevated Dissolved Oxygen and Turbidity measurements are due to naturally occurring conditions, then only the first four (4) items are required to be submitted in future reports. However, if the Department cannot determine if the Dissolved Oxygen or Turbidity is elevated due to naturally occurring conditions, then in addition to the first four (4) items, a description of the conditions at the site that caused the affected parameter(s) to be high is required to be submitted in future reports.

3.6 If the stabilization parameters in either section 3.1 or 3.2 cannot be met, and all attempts have been made to minimize the drawdown, check the instrument condition and calibration, purging flow rate and all tubing connections to determine if they might be affecting the ability to achieve stable measurements. All measurements that were made during the attempt must be documented. The sampling team leader may decide whether or

not to collect a sample or to continue purging after five (5) well volumes (conventional purge section 2.1 or 2.3 above) or five (5) volumes of the screened interval (minimizing purge volumes in section 2.2 above).

Further, the report in which the data are submitted must include the following, as applicable, except that the last four (4) items only need to be submitted once:

- Purging rate.
- Pump or tubing intake placement.
- Length and location of the screened interval.
- Drawdown in the well, if any.
- A description of conditions at the site that caused the dissolved oxygen to be high and/or dissolved oxygen measurements made within the screened or open borehole portion of the well with a downhole dissolved oxygen probe.
- A description of conditions at the site that caused the turbidity to be high and any procedures that will be used to minimize turbidity in the future.
- A description of the process and the data used to design the well.
- The equipment and procedure used to install the well.
- The well development procedure.
- Pertinent lithologic or hydrogeologic information.

If from review of the submitted data the DEP determines that both the elevated Dissolved Oxygen and Turbidity measurements are due to naturally occurring conditions, then only the first four (4) items are required to be submitted in future reports. However, if the DEP cannot determine if the Dissolved Oxygen or Turbidity is elevated due to naturally occurring conditions, then in addition to the first four (4) items, a description of the conditions at the site that caused the affected parameter(s) to be high is required to be submitted in future reports.

3.7 One fully dry purge (not recommended). This criterion applies only if purging was attempted per FS 2212, FS 2213, and section 3.4.1 below, and if it is impossible to balance the pumping rate with the rate of recharge at very low pumping rates (< 100 mL/minute).

3.7.1 If wells have previously and consistently purged dry, when purged according to FS 2212 and FS 2213, and the current depth to groundwater indicates that the well will purge dry during the current sampling event, minimize the amount of water removed from the well by using the same pump to purge and collect the sample:

3.7.1.1 Place the pump or tubing intake within the well screened interval.

3.7.1.2 Use very small diameter Teflon, Polyethylene or PP tubing and the smallest possible pump chamber volume to minimize the total volume of water pumped from the well and to reduce drawdown.

3.7.1.3 Select tubing that is thick enough to minimize oxygen transfer through the tubing walls while pumping.

3.7.1.4 Pump at the lowest possible rate (100 mL/minute or less) to reduce drawdown to a minimum.

3.7.1.5 Purge at least two (2) volumes of the pumping system (pump, tubing and flow cell, if used).

3.7.1.6 Measure pH, Specific Conductance, Temperature, Dissolved Oxygen and Turbidity and begin to collect the samples (see FS 2222).

4. Collect samples immediately after purging is complete.

4.1 The time period between completing the purge and sampling cannot exceed six (6) hours.

4.2 If sample collection does not occur within one (1) hour of purging completion, re-measure the five (5) field parameters Temperature, pH, Specific Conductance, Dissolved Oxygen and Turbidity just prior to collecting the sample.

4.2.1 If the measured values are not within 10 percent of the previous measurements, re-purge the well.

4.2.2 See section 3.4 above when collecting samples from wells that have purged dry.

FS 2213 *Purging Wells Without Plumbing (Monitoring Wells)*

1. TUBING/PUMP PLACEMENT

1.1 Do not lower the pump or intake hose (tubing) to the bottom of the well. Pump or tubing placement procedures will be determined by the purging option selected in FS 2212, section 2 above or FS 2214 below.

1.1.1 Minimizing Purge Volume: If the following conditions can be met, position the intake hose (tubing) or pump in the screened or open borehole interval.

- The same pump must be used for both purging and sampling,
- The well screen or borehole interval must be less than or equal to 10 feet, and
- The well screen or borehole must be fully submerged.

1.1.2 If the position or length of the screened interval or open borehole is unknown or estimated, place the intake hose (tubing) or pump to perform conventional purging in 1.2 below.

1.1.3 Position the pump or intake hose when purging large-diameter deep wells with open boreholes using the procedure in FS 2214 below.

1.2 Conventional Purging: Position the pump or intake tubing in the top one foot of the water column or no deeper than necessary for the type of pump.

1.2.1 If purging with a bailer, see section 4 below.

1.3 Partially Submerged Screened Interval: If the well screen or open borehole is partially submerged, and the pump will be used for both purging and sampling, position the pump or intake hose (tubing) in the portion of the water column within the submerged screened or open borehole interval.

1.3.1 If the position or length of the screened interval or open borehole is unknown or estimated, place the intake hose (tubing) or pump to perform conventional purging in 1.2 above.

1.3.2 Purge large-volume, high-recharge wells as in FS 2214 below.

1.3.3 If purging with a bailer, see section 4 below.

2. NON-DEDICATED (PORTABLE) PUMPS

2.1 Variable Speed Peristaltic Pump

2.1.1 Install a new, 1-foot maximum length of silicone tubing in the peristaltic pump head.

2.1.2 Attach a short section of tubing to the discharge side of the pump-head silicone tubing and into a graduated container.

2.1.3 Attach one end of a length of new or precleaned transport tubing to the intake side of the pump head silicone tubing.

2.1.4 Place the transport tubing in the monitoring well per one of the options in FS 2213, section 1 above.

2.1.5 Measure the depth to groundwater at frequent intervals.

2.1.6 Record these measurements.

2.1.7 Adjust the purging rate so that it is equivalent to the well recovery rate to minimize drawdown.

2.1.8 If the purging rate exceeds the well recovery rate, reduce the pumping rate to balance the withdrawal rate with the recharge rate.

2.1.9 If the water table continues to drop during pumping, lower the tubing at the approximate rate of drawdown so that the water is removed from the top of the water column.

2.1.10 Record the purging rate each time the rate changes.

2.1.11 Measure the purge volume by one of the methods outlined in FS 2212, section 1.

2.1.12 Record this measurement.

2.1.13 Decontaminate the pump and tubing between wells (see FC 1000) or only the pump if precleaned tubing is used for each well.

2.2 Variable Speed Centrifugal Pump

2.2.1 Position fuel powered equipment **downwind** and at least 10 feet from the well head. Make sure that the exhaust faces downwind.

2.2.2 Place the decontaminated suction hose so that water is always pumped from the top of the water column.

2.2.3 Equip the suction hose with a foot valve to prevent purge water from re-entering the well.

2.2.4 Measure the depth to groundwater at frequent intervals.

2.2.5 Record these measurements.

2.2.6 Adjust the purging rate so that it is equivalent to the well recovery rate to minimize drawdown.

2.2.7 If the purging rate exceeds the well recovery rate, reduce the pumping rate to balance the withdrawal rate with the recharge rate.

2.2.8 If the water table continues to drop during pumping, lower the tubing at the approximate rate of drawdown so that the water is removed from the top of the water column.

2.2.9 Record the purging rate each time the rate changes.

2.2.10 Measure the purge volume by one of the methods outlined in FS 2212, section 1.

2.2.11 Record this measurement.

2.2.12 Decontaminate the pump and tubing between wells (see FC 1000) or only the pump if precleaned tubing is used for each well.

2.3 Variable Speed Electric Submersible Pump

2.3.1 Position fuel powered equipment downwind and at least 10 feet from the well head. Make sure that the exhaust faces downwind.

2.3.2 Carefully position the decontaminated pump per one of the options in FS 2213, section 1 above.

2.3.3 Measure the depth to groundwater at frequent intervals.

2.3.4 Record these measurements.

2.3.5 Adjust the purging rate so that it is equivalent to the well recovery rate to minimize drawdown.

2.3.6 If the purging rate exceeds the well recovery rate, reduce the pumping rate to balance the withdrawal rate with the recharge rate.

2.3.7 If the water table continues to drop during pumping, lower the tubing or pump at the approximate rate of drawdown so that the water is removed from the top of the water column.

2.3.8 Record the purging rate each time the rate changes.

2.3.9 Measure the purge volume by one of the methods outlined in FS 2212, section 1.

2.3.10 Record this measurement.

2.3.11 Decontaminate the pump and tubing between wells (see FC 1000) or only the pump if precleaned tubing is used for each well.

2.4 Variable Speed Bladder Pump

2.4.1 Position fuel powered equipment **downwind** and at least 10 feet from the well head. Make sure that the exhaust faces downwind.

2.4.2 Attach the tubing and carefully position the pump per one of the options in FS 2213, section 1 above.

2.4.3 Measure the depth to groundwater at frequent intervals.

2.4.4 Record these measurements.

2.4.5 Adjust the purging rate so that it is equivalent to the well recovery rate to minimize drawdown.

2.4.6 If the purging rate exceeds the well recovery rate, reduce the pumping rate to balance the withdrawal rate with the recharge rate.

2.4.7 If the water table continues to drop during pumping, lower the tubing or pump at the approximate rate of drawdown so that the water is removed from the top of the water column.

2.4.8 Record the purging rate each time the rate changes.

2.4.9 Measure the purge volume by one of the methods outlined in FS 2212, section 1.

2.4.10 Record this measurement.

2.4.11 Decontaminate the pump and tubing between wells (see FC 1000) or only the pump if precleaned tubing is used for each well.

3. DEDICATED PORTABLE PUMPS: Place dedicated pumps per one of the options in FS 2213, section 1 above.

3.1 Variable Speed Electric Submersible Pump

3.1.1 Position fuel powered equipment **downwind** and at least 10 feet from the well head. Make sure that the exhaust faces downwind.

3.1.2 Measure the depth to groundwater at frequent intervals.

3.1.3 Record these measurements.

3.1.4 Adjust the purging rate so that it is equivalent to the well recovery rate to minimize drawdown.

3.1.5 If the purging rate exceeds the well recovery rate, reduce the pumping rate to balance the withdrawal with the recharge rate.

3.1.6 Record the purging rate each time the rate changes.

3.1.7 Measure the purge volume by one of the methods outlined in FS 2212, section 1.

3.1.8 Record this measurement.

3.2 Variable Speed Bladder Pump

3.2.1 Position fuel powered equipment **downwind** and at least 10 feet from the well head. Make sure that the exhaust faces downwind.

3.2.2 Measure the depth to groundwater at frequent intervals.

3.2.3 Record these measurements.

3.2.4 Adjust the purging rate so that it is equivalent to the well recovery rate to minimize drawdown.

3.2.5 If the purging rate exceeds the well recovery rate, reduce the pumping rate to balance the withdrawal with the recharge rate.

3.2.6 Record the purging rate each time the rate changes.

3.2.7 Measure the purge volume by one of the methods outlined in FS 2212, section 1.

3.2.8 Record this measurement.

4. BAILERS: DEP recommends against using bailers for purging except as a last contingency, or if free product is present in the well or suspected to be in the well. However, they may be used if approved by a DEP program, or specified in a permit, contract or DEP order (see Table FS 2200-3 and FS 2211, section 1.3). If in doubt about the appropriateness of using a bailer at a site or during a particular sampling event, contact the appropriate DEP program or project manager.

4.1 Minimize handling the bailer as much as possible.

4.1.1 Remove the bailer from its protective wrapping just before use.

4.1.2 Attach a lanyard of appropriate material (see FS 2201, section 4).

4.1.3 Use the lanyard to move and position the bailer.

4.2 Lower and retrieve the bailer slowly and smoothly.

4.3 Lower the bailer carefully into the well to a depth approximately a foot above the water column.

4.3.1 Do not lower the top of the bailer more than one (1) foot below the top of the water table so that water is removed from the top of the water column. Ensure that the length of the bailer does not exceed the length of the water column.

4.3.2 Allow time for the bailer to fill with aquifer water as it descends into the water column.

4.4 Carefully raise the bailer.

4.4.1 Retrieve the bailer at the same rate of 2 cm/sec until the bottom of the bailer has cleared to top of the water column.

4.5 Measure the purge volume by one of the methods outlined in FS 2212, section 1.

4.5.1 Record the volume of the bailer.

4.6 Continue to carefully lower and retrieve the bailer as described above until the purging completion conditions specified in FS 2212, section 3, have been satisfied.

4.6.1 Remove at least one (1) well volume before collecting measurements of the field parameters. Take each subsequent set of measurements after removing at least one quarter (1/4) well volume between measurements.

FS 2214 *Purging Large-Volume, High-Recharge Wells With Portable Pumps*

If a well originally constructed for high-flow-rate pumping will be sampled as a monitoring well, use these guidelines to develop a purging procedure applicable to the specific details of the well construction. Typical wells constructed for this purpose may be deep, large-diameter wells with a section of open borehole. Evaluate each well on a case-by-case basis and consider any available information on the construction and hydraulic performance of the well.

1. PURGING PROCEDURE

1.1 Place the pump at the top of the open borehole segment of the well.

1.2 Start purging while monitoring stabilization parameters as in FS 2212, section 3 above.

1.3 Purge at least one equipment volume before measuring stabilization parameters.

1.4 If the well is being purged for the first time using these guidelines, monitor stabilization parameters for an extended period until confident that sufficient volume has been pumped from the open borehole to draw fresh formation water into the pump tubing and flow-through container. Use the information obtained from the first-time purging of the well to determine the pumping rate and duration of purging required for future sampling events at the well.

1.5 Purge at least three equipment volumes before evaluating purging completion.

2. PURGING COMPLETION

2.1 Complete the purging of the well when the last three consecutive measurements of the purge stabilization parameters have met the applicable criteria specified in FS 2212, section 3 above.

3. Collect samples from the well using the procedures in FS 2221, section 1 below.

FS 2215. *Purging Wells With Plumbing (production wells or permanently installed pumps equipped with sampling ports or sampling spigots)*

Wells with in-place plumbing are commonly found at municipal water treatment plants, industrial water supplies, private residences, etc. Depending on the sampling objective for collecting samples using installed plumbing, purge the system and collect samples closest to the point of consumption, or, as close to the source well as possible. When purging is required and the purge volume of the plumbing system is not known, purge the system until the purging completion criteria in FS 2212, section 3, have been met.

1. CONTINUOUSLY RUNNING PUMPS

1.1 Select the spigot that is closest to the pump and before any storage tanks (if possible).

1.2 Remove all hoses, aerators and filters (if possible).

1.3 Open the spigot and purge at maximum flow.

1.4 If a storage tank is located between the pump and the spigot, purge the volume of the tank, lines and spigot.

1.5 If the spigot is before any storage tank, purge until sufficient volume is removed to flush the stagnant water from the spigot and the tap line to the spigot.

1.6 Reduce the flow rate to $\leq$ 500 mL/minute (a 1/8" stream) or approximately 0.1 gal/minute before collecting samples. When sampling for volatile organic compounds, reduce the flow to $\leq$100 mL/minute before collecting the samples.

2. INTERMITTENTLY RUNNING PUMPS

2.1 Select the spigot that is closest to the pump and before any storage tanks (if possible).

2.2 Remove all hoses, aerators and filters (if possible).

2.3 Open the spigot and purge sufficient volume at a maximum, practical flow rate to flush the spigot and lines and until the purging completion criteria in FS 2212, section 3, have been met.

2.4 If a storage tank is located between the pump and the spigot, purge the volume of the tank, lines and spigot.

2.5 Ensure that the purge stabilization measurement of dissolved oxygen is not biased with aeration of the sample by a high flow rate in the flow-through container.

2.6 Reduce the flow rate to $\leq$ 500 mL/minute (a 1/8" stream) or approximately 0.1 gal/minute before collecting samples. When sampling for volatile organic compounds, reduce the flow to $\leq$ 100 mL/minute before collecting the samples.

FS 2216. *Purging Airstrippers and Remedial Treatment Systems*

If collecting samples for groundwater contamination monitoring, follow FS 2215above.

FS 2220. GROUNDWATER SAMPLING TECHNIQUES

1. Purge wells using the techniques outlined in FS 2210.

2. Replace the protective covering around the well if it is soiled or torn after completing the purging operations.

3. EQUIPMENT CONSIDERATIONS

Follow all notes and restrictions as indicated in Table FS 2200-1 and as discussed in FS 2201.

NOTE: The only pumps that are currently approved for use in collecting volatile organic samples through the pump are stainless steel and Teflon variable speed submersible pumps, stainless steel and Teflon or Polyethylene variable speed bladder pumps, and permanently installed PVC bodied pumps (variable speed bladder or submersible pumps) as long as the pump remains in contact with the water in the well at all times.

3.1 Collect the sample into the sample container from the sampling device. **Do not** use intermediate containers.

3.2 In order to avoid contaminating the sample or loss of analytes from the sample:

3.3 Handle the sampling equipment as little as possible.

3.3.1 Minimize the equipment that is exposed to the sample.

3.3.2 Minimize aeration of samples collected for VOC analysis.

3.3.3 Reduce sampling pump flow rates to ≤ 100 mL/minute when collecting VOC samples.

3.4 Dedicated Sampling Equipment

3.4.1 Whenever possible, use dedicated equipment because it significantly reduces the chance of cross-contamination.

3.4.2 Dedicated is defined as equipment that is to be used solely for one location for the life of that equipment (e.g., permanently mounted pump).

3.4.3 All material construction and restrictions from Table FS 2200-1 also apply to dedicated equipment. Purchase equipment with the most sensitive analyte of interest in mind.

3.5 Cleaning/Decontamination

3.5.1 Clean or ensure dedicated pumps are clean before installation. They do not need to be cleaned prior to each use but must be cleaned if they are withdrawn for repair or servicing.

3.5.2 Clean or make sure any permanently mounted tubing is clean before installation.

3.5.3 Change or clean tubing when the pump is withdrawn for servicing.

3.5.4 Clean any replaceable or temporary parts as specified in FC 1000.

3.5.5 Collect equipment blanks on dedicated pumping systems when the tubing is cleaned or replaced.

3.5.6 Clean or ensure dedicated bailers are clean before placing them into the well.

3.5.7 Collect an equipment blank on dedicated bailers before introducing them into the water column.

3.5.8 Suspend dedicated bailers above the water column if they are stored in the well.

FS 2221. *Sampling Wells Without Plumbing*

1. SAMPLING WITH PUMPS: Variable speed stainless steel and Teflon submersible pumps and stainless steel, Teflon or Polyethylene bladder pumps, and permanently installed PVC-bodied variable speed submersible or bladder pumps, as long as the pump remains in contact with the water in the well at all times, may be used to sample for all organics. The delivery tubing must be Teflon, Polyethylene or PP. **Extractable organics** may be collected through a peristaltic pump if ≤ 1 foot of silicone tubing is used in the pump head or a vacuum trap is used (see Figure FS 2200-1 for specific configuration). Follow all notes and restrictions as defined in Table FS 2200-1 and discussed in Equipment and Supplies (FS 2201) when using pumps to collect samples.

Do not lower the pump or tubing to the bottom of the well.

1.1 Peristaltic Pump

1.1.1 Volatile Organics Using Manual Fill and Drain Method: Collect volatile organics last. If the pump tubing is placed within the screened interval, the tubing cannot be reinserted into the well, and steps 1.1.1.3 through 1.1.1.6 below are prohibited.

1.1.1.1 Ensure that there is sufficient tubing volume to fill the requisite number of VOC vials.

1.1.1.2 Remove the drop tubing from the inlet side of the pump.

1.1.1.3 Submerse the drop tubing into the water column and allow it fill.

1.1.1.4 Remove the drop tubing from the well.

1.1.1.5 Prevent the water in the tubing from flowing back into the well.

1.1.1.6 Carefully allow the groundwater to drain by gravity into the sample vials. Avoid turbulence. Do not aerate the sample. The flow rate must be ≤ 100 mL/minute.

1.1.1.7 Repeat steps 1.1.1.3 - 1.1.1.6 until enough vials are filled.

1.1.2 Volatile Organics Using the Pump to Fill and Drain the Tubing: Collect volatile organics last. If the pump tubing is placed within the screened interval, the tubing cannot be reinserted into the well, and steps 1.1.2.2 through 1.1.2.8 below are prohibited.

1.1.2.1 Ensure that there is sufficient tubing volume to fill the requisite number of VOC vials.

1.1.2.2 Submerse the drop tubing into the water column.

1.1.2.3 Use the pump to fill the drop tubing.

1.1.2.4 Quickly remove the tubing from the pump.

1.1.2.5 Prevent the water in the tubing from flowing back into the well.

1.1.2.6 Remove the drop tubing from the well and fill the vials using the pump or gravity-drain methods in steps 1.1.2.7 or 1.1.2.8 below.

1.1.2.7 Reverse the flow on the peristaltic pump to deliver the sample into the vials at a slow, steady rate. The flow rate must be ≤ 100 mL/minute.

1.1.2.8 Or, remove the drop tubing from the inlet side of the pump and carefully allow the groundwater to drain into the sample vials. Avoid turbulence. Do not aerate the sample. The flow rate must be ≤ 100 mL/minute.

1.1.2.9 Repeat steps 1.1.2.2 through 1.1.2.8 until enough vials are filled.

1.1.3 Extractable Organics Collected Through Silicone Pump-Head Tubing:

1.1.3.1 Ensure that a 1-foot maximum length of new silicone tubing was installed in the peristaltic pump head assembly before the well was purged if the same pump is being used to purge and sample the well. Otherwise, install a new length of tubing as described above.

1.1.3.2 Collect extractable organic samples directly from the effluent delivery tubing (attached to discharge side of the silicone pump head tubing) into the sample container.

1.1.3.3 If there is a concern that sample analytes are absorbed, adsorbed, leached or otherwise affected or lost by pumping through the silicone pump-head tubing, sample the well using the organic trap assembly in 1.1.4 below.

1.1.4 Extractable Organics Using an Optional Organic Trap Assembly

1.1.4.1 Assemble the components of the pump and trap according to Figure FS 2200-1.

1.1.4.2 The sample container should be the trap bottle.

1.1.4.3 All equipment that contacts the groundwater **before** the sample container must be constructed of Teflon, Polyethylene, PP, stainless steel or glass, including the transport tubing to and from the sample container, the interior liner of the container cap and all fittings. **Do not use a rubber stopper as a cap.**

1.1.4.4 Connect the outflow tubing from the container to the influent side of the peristaltic pump.

1.1.4.5 Prevent the water in the down-hole delivery tubing from flowing back into the well while performing this connection.

1.1.4.6 Turn the pump on and reduce the flow rate to a smooth and even flow.

1.1.4.7 Discard a small portion of the sample to allow an air space.

1.1.4.8 Preserve (if required), label and complete the field notes.

1.1.5 Inorganics

1.1.5.1 Inorganic samples may be collected from the effluent tubing.

1.1.5.2 If samples are collected from the pump, decontaminate all tubing (including the tubing in the head) or change it between wells.

1.1.5.3 Preserve (if required), label and complete field notes.

1.2 Variable Speed Bladder Pump

1.2.1 If sampling for organics the pump body must be constructed of stainless steel and the valves and bladder must be Teflon. All tubing must be Teflon, Polyethylene, or PP and any cabling must be sealed in Teflon, Polyethylene or PP, or made of stainless steel.

1.2.2 After purging to a smooth even flow, reduce the flow rate.

1.2.3 When sampling for volatile organic compounds, reduce the flow rate to 100 mL/minute or less, if possible.

1.3 Variable Speed Submersible Pump

1.3.1 The housing must be stainless steel.

1.3.2 If sampling for organics, the internal impellers, seals and gaskets must be constructed of stainless steel, Teflon, Polyethylene or PP. The delivery tubing must be Teflon, Polyethylene or PP and the electrical cord must be sealed in Teflon and any cabling must be sealed in Teflon or constructed of stainless steel.

1.3.3 After purging to a smooth even flow, reduce the flow rate.

1.3.4 When sampling for volatile organic compounds, reduce the flow rate to 100 mL/minute or less, if possible.

2. SAMPLING WITH BAILERS: A high degree of skill and coordination are necessary to collect representative samples with a bailer. When properly used, bailers may be used to collect samples for certain analyte groups and under specific conditions (see Table FS 2200-3). They must be of an appropriate type and construction (see FS 2201, section 3), and must be used as outlined below. If in doubt about the appropriateness of using a bailer at a site or during a particular sampling event, contact the appropriate DEP program or project manager.

2.1 General Considerations

2.1.1 Minimize handling the bailer as much as possible.

2.1.1.1 Wear sampling gloves.

2.1.1.2 Remove the bailer from its protective wrapping just before use.

2.1.1.3 Attach a lanyard of appropriate material (see FS 2201, section 4).

2.1.1.4 Use the lanyard to move and position the bailers.

2.1.2 Do not allow the bailer or lanyard to touch the ground.

2.1.3 Rinsing

2.1.3.1 If the bailer is certified precleaned, no rinsing is necessary.

2.1.3.2 If both a pump and a bailer are to be used to collect samples, rinse the exterior and interior of the bailer with sample water from the pump before removing the pump.

2.1.3.3 If the purge pump is not appropriate for collecting samples (e.g., non-inert components), rinse the bailer with by collecting a single bailer of the groundwater to be sampled. Use the technique described in section 2.2, Bailing Technique, below.

2.1.3.4 Discard the water appropriately.

2.1.3.5 **Do not** rinse the bailer if Oil & Grease, TRPHs, etc., (see FS 2006) are to be collected.

2.2 Bailing Technique

2.2.1 Collect all samples that are required to be collected with a pump before collecting samples with the bailer.

2.2.2 Raise and lower the bailer gently to minimize stirring up particulate matter in the well and the water column which can increase sample turbidity.

2.2.3 Lower the bailer carefully into the well to a depth approximately a foot above the water column. Ensure that the length of the bailer does not exceed the length of the water column.

2.2.3.1 When the bailer is in position, lower the bailer into the water column at a rate of 2 cm/sec until the desired depth is reached (see section 2.2.3 above).

2.2.4 Do not lower the top of the bailer more than one (1) foot below the top of the water table so that water is removed from the top of the water column.

2.2.5 Allow time for the bailer to fill with aquifer water as it descends into the water column.

2.2.6 Do not allow the bailer to touch the bottom of the well or particulate matter will be incorporated into the sample.

2.2.6.1 Carefully raise the bailer (see section 2.2.2 above). Retrieve the bailer at the same rate of 2 cm/sec until the bottom of the bailer has cleared to top of the water column.

2.2.7 Lower the bailer to approximately the same depth each time.

2.2.8 Collect the sample.

2.2.8.1 Install a device to control the flow from the bottom of the bailer and discard the first few inches of water. Reduce the flow to $\leq$ 100 mL/minute when collecting VOC samples.

2.2.8.2 Fill the appropriate sample containers by allowing the sample to slowly flow down the side of the container. Minimize aeration of VOC samples.

2.2.8.3 Discard the last few inches of water in the bailer.

2.2.9 Repeat steps 2.2.1 through 2.2.8.3 for additional samples.

2.2.10 Measure the DO, pH, temperature, turbidity and specific conductance after the final sample has been collected.

2.2.10.1 Record all measurements and note the time that sampling was completed.

3. SAMPLING WELLS WITH FLOATING NON-AQUEOUS PHASE LIQUID: DEP does not recommend the sampling of wells with floating non-aqueous phase liquid for trace contaminants. This concerns primarily petroleum related sites, but includes any chemical product (e.g., solvent) that floats on the water table. Sampling is acceptable if the information is to be used for the purpose of remedial design.

Sample data from such wells cannot provide useful information regarding the level of contamination. Furthermore, these wells typically do not provide legitimate data because of permanent chemical contamination from product contact with the well casing for an extended period of time.

DEP does reserve the right to require sampling of these wells, not for levels of trace contaminants, but for confirmation of an appropriate remediation technique. This type of sampling is performed **below** the non-aqueous phase layer (see section 3.2 below).

3.1 Non-Aqueous Phase Liquid Sampling: Non-aqueous phase liquid may be evident in a cased monitoring well or in an open excavation.

3.1.1 Non-aqueous phase liquid is normally sampled for two reasons:

- Documentation for its existence and thickness; and
- Determination of the type of product so that the proper analyses can be performed to determine extent. This is only feasible for relatively recent releases as it may not be possible to identify weathered product.

3.1.2 Disposable plastic (acrylic, clear PVC) bailers are recommended for sampling. Disposable Polyethylene and PP bailers are also acceptable. Other wide mouth vessels may be used for sampling non-aqueous phase liquid in an excavation.

3.1.3 Monitoring Well

3.1.3.1 If a non-aqueous phase liquid is identified in a monitoring well during the water level measurement, measure its thickness in the well. If the thickness of the non-aqueous phase liquid is greater than 0.01 foot or product globules are present, collect a sample using a precleaned disposable bailer.

3.1.3.2 Measure the product thickness to the nearest 0.01 foot after withdrawing the bailer.

3.1.3.3 Pour a portion of the product into a glass sample container.

3.1.3.4 This sample is considered a concentrated waste. Therefore, package the container in protective wrapping to prevent breakage, isolate from other samples, and ice to 4°C.

3.1.4 Excavation

3.1.4.1 If non-aqueous phase liquid is observed in an open excavation, a glass sample container or a precleaned intermediate vessel may be used to collect the sample.

3.1.4.2 Securely tie a lanyard to the container and lower it into the excavation.

3.1.4.3 Gently lower and retrieve the container so that no solid material is released or collected.

3.1.4.4 If sufficient water is available, a bailer can be used.

3.1.4.5 Although not recommended, screened casing can be placed (or augered and placed) in the bottom of the excavation and the product sampled with a bailer.

3.1.4.6 Avoid dangerous situations, such as standing too close to the edge of an excavation, riding in the backhoe bucket, or entering a trench or excavation that may collapse.

3.1.4.7 Follow all applicable OSHA regulations.

3.2 Sampling Below Product

3.2.1 This type of depth-specific sampling to attempt to sample the dissolved constituents in the water column below the product layer is performed only at the request of DEP or its designee.

3.2.2 These data provide information that helps define adequate groundwater treatment. Without these data, incorrect (and sometimes unnecessarily expensive) remediation techniques may be designed for a situation where they are not required.

3.2.3 There are some substantial logistical problems involved with sending a sampler through non-aqueous phase liquid to sample the groundwater below. Although there are some products designed specifically for this type of sampling, they are expensive and the results may not be commensurate with their cost. The use of "self-engineered" equipment or coverings may be the best option.

3.2.4 These data are only to be used for qualitative use and will aid in deciding on an appropriate remediation technique.

3.2.5 Wrapping bailers and tubing in plastic seems to be the most popular technique in getting past the product layer.

3.2.6 Although not recommended, some have wrapped submersible pumps in several layers of plastic and retrieved each layer by a separate lanyard. One suggestion would be to use a rigid piece of stainless steel tubing wrapped in plastic.

3.2.6.1 Once the covered tubing is past the layer, pull up on the plastic, piercing the plastic and exposing the (somewhat) clean tubing inlet.

3.2.6.2 Introduce the wrapped hose slowly to not entrain any more product into the dissolved layer located below.

3.2.6.3 Also, perform this procedure with a peristaltic pump or a vacuum pump linked to a trap bottle. To use this setup, the water table must be no deeper than 15-20 feet, realizing that actual sampling may be occurring several feet below the product layer.

FS 2222. *Sampling Low Permeability Aquifers or Wells That Have Purged Dry*

1. Collect the sample(s) after the well has been purged according to FS 2212, section 3.4. Minimize the amount of water removed from the well by using the same pump to purge and collect the sample. If the well has purged dry, collect samples as soon as sufficient sample water is available.

2. Measure the five (5) field parameters Temperature, pH, Specific Conductance, Dissolved Oxygen and Turbidity at the time of sample collection.

3. Advise the analytical laboratory and the client that the usual amount of sample for analysis may not be available.

FS 2223. *Sampling Wells With In-Place Plumbing*

1. If a storage tank is present, locate a cold water spigot, valve or other sampling point close to the well head between the pump and the storage tank. If there is no sampling location between the pump and the storage tank, locate the spigot, valve or other sampling point closest to the tank.

1.1 Depending on the sampling objective for collecting samples using installed plumbing, purge the system and collect samples closest to the point of consumption, or, as close to the source well as possible.

2. Remove all screens or aerators and reduce the flow rate to no more than 500 mL/minute. If collecting samples for volatile organic compounds, reduce the flow rate to 100 mL/minute or less. Collect the samples directly into the appropriate containers.

FS 2224. *Sampling Airstripper and Remedial Treatment System Sampling*

1. Reduce the flow rate to less than 500 mL/minute and begin sample collection.

2. If collecting samples for volatile organic compounds, reduce the flow rate to 100 mL/minute or less.

3. Collect the samples directly into the appropriate containers.

FS 2225. *Filtering Groundwater Samples*

Filtered groundwater samples can only be collected after approval from the DEP program or project manager. If filtering is approved, the DEP program or permit condition may require both filtered and unfiltered samples to be collected, analyzed and reported.

1. FILTERING GROUNDWATER FOR METALS:

1.1 Unless specified otherwise by the DEP program, use a new, disposable, high capacity, 1-µm in-line filter.

1.2 Use a variable speed peristaltic, bladder or submersible pump with the in-line filter fitted on the outlet end.

1.2.1 Peristaltic pumps, bladder pumps or submersible pumps can be used when water levels are no greater than 20 to 25 feet deep.

1.2.2 Bladder pumps or submersible pumps must be used when water levels are greater than 20 to 25 feet deep.

1.3 Ensure that a 1-foot maximum length of new, silicone tubing was installed in the peristaltic pump head assembly before the well was purged if the same pump is being used to purge and sample the well. Otherwise, install a new length of tubing as described above.

1.4 Ensure that new or precleaned delivery tubing was assembled with the peristaltic pump before the well was purged if the same pump is being used to purge and sample the well. Otherwise, assemble the pump with new or precleaned delivery tubing and the new filter.

1.5 Insert the filter on the high pressure side (i.e., on the delivery side) of the pump.

1.5.1 Flush the filter before attaching to the pump tubing assembly with 30-50 mL of analyte free water or an inert gas (nitrogen) to remove atmospheric oxygen;

1.5.2 Or, with the filter attached to the pump tubing assembly, hold the filter upright with the inlet and outlet in the vertical position and pump water from the aquifer through the filter until all atmospheric oxygen has been removed.

1.6 Collect the filtered samples directly into the sample container from the high-pressure (delivery) side of the pump tubing assembly.

1.6.1 Collect filtered samples by either of the methods in 1.6.1.3 or 1.6.1.4 below if the static water level in the well is too deep for a variable speed peristaltic pump and a variable speed electric submersible pump or variable speed bladder pump is not available.

1.6.1.1 Do not agitate the sample or expose it to atmospheric oxygen.

1.6.1.2 **Do not** pour the sample into any intermediate vessel for subsequent filtration.

1.6.1.3 Collect the sample in a Polyethylene, Teflon or PP bailer that can be pressurized. When the bailer has been retrieved, immediately connect the filter and begin to pressurize the bailer;

1.6.1.4 Or, collect the sample with a bailer and immediately place the intake tube of the peristaltic pump into the full bailer and begin pumping the water through the filter as described in section 1.2 above.

1.7 **Do not** use the following equipment for filtering groundwater samples for metals:

1.7.1 Any pump and apparatus combination in which the filter is on the vacuum (suction) side of the pump.

1.7.2 Any type of syringe or barrel filtration apparatus.

1.7.3 Any filter that is not encased in a one-piece, molded unit.

2. Filtering groundwater for non-metallic analytes

2.1 The following analytes cannot be filtered:

- Oil and Grease
- Total Recoverable Petroleum Hydrocarbons (TRPH)
- FL-PRO
- Volatile Organic Compounds (VOC)
- Microbiological Analytes
- Volatile Inorganic Compounds (e.g., Hydrogen Sulfide)

2.2 Unless specified otherwise by the regulatory program, use a new, disposable, high capacity, 0.45 µm in-line filter.

2.3 Assemble the pump, tubing and filter as in 1.2 – 1.5 above.

2.4 Flush the filter as in 1.5.1 or 1.5.2 above.

2.5 Collect the samples as in 1.6 – 1.6.1.4 above.

Appendix FS 2200

Tables, Figures and Forms

Table FS 2200-1
Equipment for Collecting Groundwater Samples

Activity	Equipment Type
Well Purging	Variable speed centrifugal pump Variable speed submersible pump Variable speed bladder pump Variable speed peristaltic pump Bailer with lanyard: Not Recommended
Well Stabilization	pH meter DO meter Conductivity meter Thermometer/Thermistor Turbidimeter Flow-through cell Multi-function meters
Sample Collection	Variable speed peristaltic pump Variable speed submersible pump Variable speed bladder pump Bailer with lanyard (See Table FS 2200-3)
Filtration	Variable speed peristaltic pump Variable speed submersible pump Variable speed bladder pump Pressurized bailer 1.0 µm high capacity molded filter 0.45 µm high capacity molded filter
Groundwater Level	Electronic sensor Chalked tape

Table FS 2200-2
Dissolved Oxygen Saturation

TEMP	D.O.	mg/L	TEMP	D.O.	mg/L	TEMP	D.O.	mg/L	TEMP	D.O.	mg/L
deg C	SAT.	20%	deg C	SAT.	20%	deg C	SAT.	20%	deg C	SAT.	20%
15.0	10.084	2.017	19.0	9.276	1.855	23.0	8.578	1.716	27.0	7.968	1.594
15.1	10.062	2.012	19.1	9.258	1.852	23.1	8.562	1.712	27.1	7.954	1.591
15.2	10.040	2.008	19.2	9.239	1.848	23.2	8.546	1.709	27.2	7.940	1.588
15.3	10.019	2.004	19.3	9.220	1.844	23.3	8.530	1.706	27.3	7.926	1.585
15.4	9.997	1.999	19.4	9.202	1.840	23.4	8.514	1.703	27.4	7.912	1.582
15.5	9.976	1.995	19.5	9.184	1.837	23.5	8.498	1.700	27.5	7.898	1.580
15.6	9.955	1.991	19.6	9.165	1.833	23.6	8.482	1.696	27.6	7.884	1.577
15.7	9.934	1.987	19.7	9.147	1.829	23.7	8.466	1.693	27.7	7.870	1.574
15.8	9.912	1.982	19.8	9.129	1.826	23.8	8.450	1.690	27.8	7.856	1.571
15.9	9.891	1.978	19.9	9.111	1.822	23.9	8.434	1.687	27.9	7.842	1.568
16.0	9.870	1.974	20.0	9.092	1.818	24.0	8.418	1.684	28.0	7.828	1.566
16.1	9.849	1.970	20.1	9.074	1.815	24.1	8.403	1.681	28.1	7.814	1.563
16.2	9.829	1.966	20.2	9.056	1.811	24.2	8.387	1.677	28.2	7.800	1.560
16.3	9.808	1.962	20.3	9.039	1.808	24.3	8.371	1.674	28.3	7.786	1.557
16.4	9.787	1.957	20.4	9.021	1.804	24.4	8.356	1.671	28.4	7.773	1.555
16.5	9.767	1.953	20.5	9.003	1.801	24.5	8.340	1.668	28.5	7.759	1.552
16.6	9.746	1.949	20.6	8.985	1.797	24.6	8.325	1.665	28.6	7.745	1.549
16.7	9.726	1.945	20.7	8.968	1.794	24.7	8.309	1.662	28.7	7.732	1.546
16.8	9.705	1.941	20.8	8.950	1.790	24.8	8.294	1.659	28.8	7.718	1.544
16.9	9.685	1.937	20.9	8.932	1.786	24.9	8.279	1.656	28.9	7.705	1.541
17.0	9.665	1.933	21.0	8.915	1.783	25.0	8.263	1.653	29.0	7.691	1.538
17.1	9.645	1.929	21.1	8.898	1.780	25.1	8.248	1.650	29.1	7.678	1.536
17.2	9.625	1.925	21.2	8.880	1.776	25.2	8.233	1.647	29.2	7.664	1.533
17.3	9.605	1.921	21.3	8.863	1.773	25.3	8.218	1.644	29.3	7.651	1.530
17.4	9.585	1.917	21.4	8.846	1.769	25.4	8.203	1.641	29.4	7.638	1.528
17.5	9.565	1.913	21.5	8.829	1.766	25.5	8.188	1.638	29.5	7.625	1.525
17.6	9.545	1.909	21.6	8.812	1.762	25.6	8.173	1.635	29.6	7.611	1.522
17.7	9.526	1.905	21.7	8.794	1.759	25.7	8.158	1.632	29.7	7.598	1.520
17.8	9.506	1.901	21.8	8.777	1.755	25.8	8.143	1.629	29.8	7.585	1.517
17.9	9.486	1.897	21.9	8.761	1.752	25.9	8.128	1.626	29.9	7.572	1.514
18.0	9.467	1.893	22.0	8.744	1.749	26.0	8.114	1.623	30.0	7.559	1.512
18.1	9.448	1.890	22.1	8.727	1.745	26.1	8.099	1.620	30.1	7.546	1.509
18.2	9.428	1.886	22.2	8.710	1.742	26.2	8.084	1.617	30.2	7.533	1.507
18.3	9.409	1.882	22.3	8.693	1.739	26.3	8.070	1.614	30.3	7.520	1.504
18.4	9.390	1.878	22.4	8.677	1.735	26.4	8.055	1.611	30.4	7.507	1.501
18.5	9.371	1.874	22.5	8.660	1.732	26.5	8.040	1.608	30.5	7.494	1.499
18.6	9.352	1.870	22.6	8.644	1.729	26.6	8.026	1.605	30.6	7.481	1.496
18.7	9.333	1.867	22.7	8.627	1.725	26.7	8.012	1.602	30.7	7.468	1.494
18.8	9.314	1.863	22.8	8.611	1.722	26.8	7.997	1.599	30.8	7.456	1.491
18.9	9.295	1.859	22.9	8.595	1.719	26.9	7.983	1.597	30.9	7.443	1.489

Derived using the formula in Standard Methods for the Examination of Water and Wastewater, Page 4-101, 18th Edition, 1992

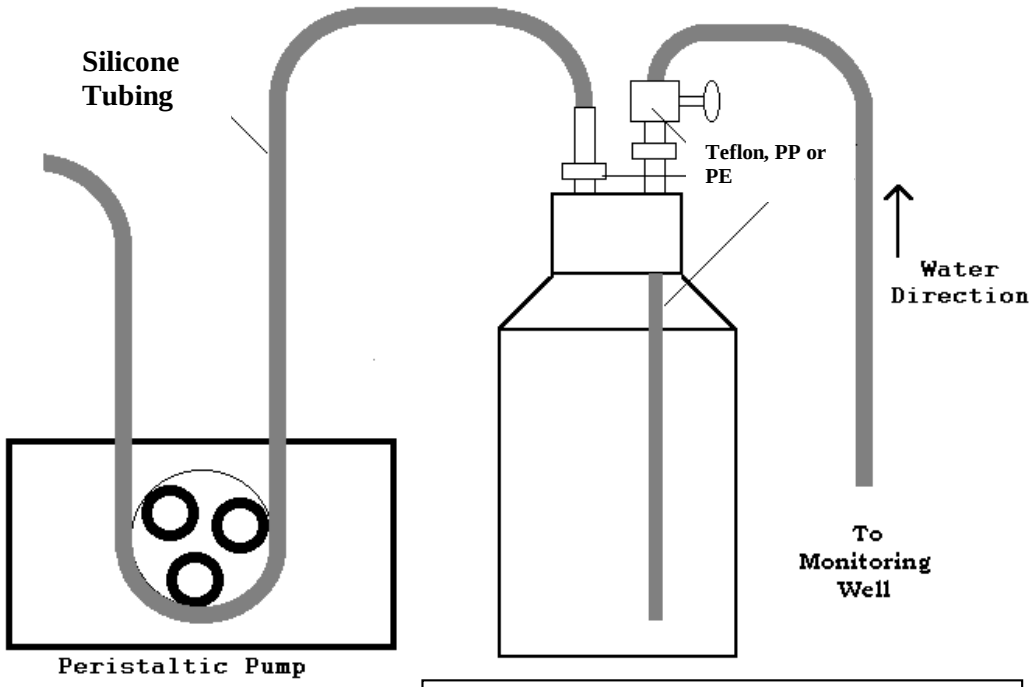
Table FS 2200-3
Allowable Uses for Bailers

• **ANALYTE GROUP(S)**	• **PURGING** (Not Recommended)	• **SAMPLING**	
	Use:	Use:	Not Recommended:
Volatile Organics Extractable Organics Radionuclides, including Radon Metals Volatile Sulfides	If allowed by permit, program, contract or order or If operated by a skilled individual with documented training in proper techniques. Field documentation must demonstrate that the procedure in FS 2213, section 4 was followed without deviation.	If concentrations exceed action levels, the purpose is to monitor effective treatment, and the DEP program allows the use of bailers; or If specified by DEP permit, program, contract or order. or If operated by a skilled individual with documented training in proper techniques and using appropriate equipment. Field documentation must demonstrate that the procedure in FS 2221, section 2 was followed without deviation.	If concentrations are near or below the stated action levels; or If a critical decision (e.g., clean closure) will be made based on the data; or If data are to demonstrate compliance with a permit or order.
Petroleum Hydrocarbons (TRPH) & Oil & Grease	If allowed by permit, program, contract or order or If operated by a skilled individual with documented training in proper techniques. Field documentation must demonstrate that the procedure in FS 2213, section 4 was followed without deviation.	Only if allowed by permit, program, contract or order as samples should be collected into the container without intermediate devices.	Unless allowed by permit, program, contract or order.

• **ANALYTE GROUP(S)**	• **PURGING** (Not Recommended)	• **SAMPLING**	
	Use:	Use:	Not Recommended:
Biologicals Inorganic Non-Metallics Aggregate Organics Microbiological Physical and Aggregate Properties	If allowed by permit, program, contract or order or If operated by a skilled individual with documented training in proper techniques. Field documentation must demonstrate that the procedure in FS 2213, section 4 was followed without deviation.	If all analytes collected from the well can be collected with a bailer; or If collected after collecting all analytes that require the use of a pump.	Before collecting any analytes that must be collected with a pump.
Ultra-Trace Metals	Never	Never	

Figure 2200-1

Pump and Trap for Extractable Organics



The glass sample bottle must be threaded to use a reusable sampling cap lined and installed with fittings made of Teflon, polypropylene or polyethylene, similar to the design shown.

Scenario 1: WELL SCREEN COMPLETELY SUBMERGED

Scenario 2: WELL SCREEN PARTIALLY SUBMERGED

Option 1a: Minimal Purge Volume: Pump or tubing is placed within the middle of the screen interval. The following conditions must be met to use this option:

1. The well screen interval is ≤ 10 feet.
2. Although drawdown may occur in the well when purging is initiated, the drawdown has to stabilize (Aquifer Recovery Rate = Purge Rate).
3. The samples will be obtained with the same equipment that was used to purge the well. Therefore, centrifugal pumps and bailers are not suitable for use in Option 1a.

If one or more of these conditions do not apply, use Option 1b.

Option 1b: Conventional Purge: Pump, tubing, or bailer [1] is placed above the screen at the top of the water column.

[1] DEP does not recommend the use of a bailer for purging; however, if a bailer is used it shall be lowered and raised at the rate of 2 cm/sec in the top of the water column.

Option 2a: A bailer [1] is placed at the top of the water column and is used to purge and sample the well.

Option 2b: Pump or tubing is placed within the middle of the saturated portion of the screen interval.

If the pump or tubing that was used for purging will not be used to obtain the sample, then position the pump or tubing at the top of the water column for purging.

Purging Procedure #1

1. After the drawdown in the well stabilizes, purge at least one equipment volume then collect the first set of stabilization parameters.
2. Thereafter, collect stabilization parameters ≥ 2 to 3 minutes apart.
3. Purge at least three equipment volumes before sampling.

Purging Procedure #2

1. Purge at least one well volume then collect first set of stabilization parameters.
2. Thereafter, collect stabilization parameters ≥ every 1/4 well volume.

Purging Procedure #3

1. Purge at least one well volume then collect first set of stabilization parameters.
2. Thereafter, collect stabilization parameters ≥ 2 to 3 minutes apart.

Purging Completion

If Dissolved Oxygen is ≤ 20% of saturation for the measured temperature and Turbidity is ≤ 20 NTUs, then purging is complete when three consecutive readings of the parameters listed below are within the following ranges:

Temperature ± 0.2 °C
pH ± 0.2 Standard Units
Specific Conductance ± 5.0% of reading

If Dissolved Oxygen (DO) is > 20% of saturation for the measured temperature and/or Turbidity is > 20 NTUs after every attempt has been made to reduce DO and/or turbidity, then purging is complete when three consecutive readings of the parameters listed below are within the following ranges:

Temperature ± 0.2 °C
pH ± 0.2 Standard Units
Specific Conductance ± 5.0% of reading
Dissolved Oxygen ± 0.2 mg/L or readings are within 10% (whichever is greater).
Turbidity ± 5 NTUs or readings are within 10% (whichever is greater).

If the well is expected to purge dry, position the pump or tubing within the screened interval and purge at ≤ 100 mL/minute until two equipment volumes are removed. Use the same pump for purging and sampling.

If the well purges dry at the lowest achievable flow rate (pumping at 100 mL/minute or less), then after a sufficient amount of water recharges in the well, collect the samples.

In either case listed above, before samples are collected, measure (once) pH, temperature, specific conductance, dissolved oxygen, and turbidity.

If one or more parameters do not stabilize after 5 volumes of the screened interval (purging procedure #1) or 5 well volumes (purging procedure #s 2 & 3) are removed, purging may be discontinued at the discretion of the sampling team leader.

FS 3000. SOIL

See also the following Standard Operating Procedures:

- FA 1000 Administrative Procedures
- FC 1000 Cleaning/Decontamination Procedures
- FD 1000 Documentation Procedures
- FM 1000 Field Planning and Mobilization
- FQ 1000 Field Quality Control Requirements
- FS 1000 General Sampling Procedures
- FT 1000 – FT 2000 Field Testing and Calibration

1. Introduction and Scope

1.1. Use these SOPs during field investigations to collect soil samples that are representative of current site conditions. It is very important to ensure that the collected samples are neither altered nor contaminated by sampling and handling techniques.

1.2. The following topics include: equipment choice, equipment construction materials, grab and areal or depth composite sampling techniques. Sample collection methods fall into three general depth classifications: surface, shallow subsurface, and deep subsurface. Once the samples are acquired, the handling procedures are very similar and are described below.

2. GENERAL

2.1. Select sampling equipment based on the type of sample to be collected and the analytes of interest. Choose soil sampling locations such that a representative portion of the soil is collected with minimal disturbance. Locations where natural vegetation is stressed or dead and/or areas that have surficial soil staining may be indicative of improper waste disposal practices.

2.2. If background and/or quality control sampling is warranted and feasible as determined in the site's work plan or by the project manager, select an up gradient, undisturbed location for obtaining the background and/or quality control samples. Be aware that differences in soil types may affect these background samples (e.g., sands vs. clays).

2.3. **Do not collect** samples for chemical analysis from auger flights or cuttings from hollow stem auger flights, except for waste characterization purposes for disposal.

2.4. Do not use samples that are collected for geological/lithological or vapor meter determinations for chemical analyses.

3. EQUIPMENT AND SUPPLIES

3.1. All equipment must be constructed of materials consistent with the analytes of interest. Refer to FS 1000, Tables FS 1000-1, FS 1000-2 and FS 1000-3 for selection of appropriate equipment and materials.

3.2. For information on sample container size and construction, see FS 1000, Table FS 1000-6.

3.3. For information on sampling equipment cleaning requirements, see FC 1000.

3.4. For information on preservation and holding time requirements, see FS 1000, Table FS 1000-6.

3.5. For information on documentation requirements, see FD 1000.

4. PROCEDURES FOR COMPOSITING

4.1. The following is not a complete discussion regarding all available sampling protocols nor the appropriateness or inappropriateness of compositing soil samples. The appropriateness of compositing soil samples will depend on the data quality objectives of the project. However, it is sometimes advantageous to composite soil samples to minimize the number of samples to be analyzed when sampling highly contaminated areas. Obtain permission from the DEP program.

4.1.1. Select sampling points from which to collect each aliquot.

4.1.2. Using the appropriate sampling technique, collect equal aliquots (same sample size) from each location and place in a properly cleaned container.

4.1.3. **Combine the aliquots of the sample directly in the sample container with no pre-mixing**.

4.1.4. Record the amount of each aliquot (volume or weight).

4.1.5. Label container, preserve on wet ice to 4°C and complete field notes.

4.1.6. Notify the laboratory that the sample is an unmixed composite sample, and request that the sample be thoroughly mixed before sample preparation or analysis.

5. SPECIFIC PROCEDURES FOR VOLATILE ORGANIC COMPOUNDS

Follow the procedures specified in EPA Method 5035 for sample collection and sample preparation. The protocols listed below **do not replace Method 5035** but clarify and/or modify certain method procedures. Therefore, it is essential that all organizations have a copy of Method 5035 as a reference document.

5.1. Container Preparation

5.1.1. All containers must be cleaned according to the FC 1000 sample container cleaning procedures for volatile organics.

5.1.2. Sample Vials: If sample vials are filled in the field, they must be provided with all reagents, stirring devices, label **and vial cap** to be used during sample analysis. These vials must be preweighed by the laboratory and records must be maintained so that there is an unambiguous link between the tare weight and the filled sample vial.

5.2. Collection Procedure

5.2.1. The sample vials (when used) will contain a premeasured amount of liquid. The laboratory must weigh the vials before sending into the field, and must weigh them again after receipt. Therefore:

- Do not lose any of the liquid either through evaporation or spillage
- Do not use a vial if some of the contents has spilled, or if it appears that some has leaked during transport
- Use the laboratory-supplied container label for identification information. **DO NOT apply any additional labels to the container**

- Do not interchange vial caps or septa

5.2.2. Minimize exposure to air by obtaining the sample directly from the sample source, using a coring device or a commercially designed sampling tool.

5.2.2.1. The sample collection device must be designed to fit tightly against the mouth of the vial or be small enough to be inserted into the vial. Use:

- EnCore or equivalent sampling devices or
- Disposable plastic syringes with the syringe end cut off prior to sampling (use **once** per sampling location).

5.2.2.2. Extrude the sample directly into the sample container.

5.2.3. Follow the method procedures for field transfer into the vial.

5.2.4. Procedures for determining the sample weight in the field are not required unless the project manager requires an accurate determination of the 5-gram sample size.

5.2.4.1. If the vials are returned to the laboratory for weighing, the sampler must be proficient in estimating the requisite 5-gram weight necessary for each sample.

5.2.4.2. If an accurate estimate of the 5-gram sample size is desired prior to starting sample collection activities, use a balance with a sensitivity of 0.1 gram. Check the balance calibration before each day's use with a set of weights that have been calibrated against NIST-traceable weights at least annually.

5.2.5. If the sampling device is transported to the laboratory with a sample, make sure the seals are intact, especially if collecting samples from sandy soils.

5.2.6. Collect at least two replicate samples from the same soil stratum and within close proximity to the original sample location.

5.2.7. Collect an additional aliquot of sample for screening and dry weight determinations.

5.3. Preservation (see FS 1000, Table FS 1000-7)

5.3.1. Low Level (≤ 200 µg/kg volatile organics)

5.3.1.1. Method 5035 discusses the use of sodium bisulfate, which is an acid. Since Florida soils contain significant amounts of calcium carbonate that reacts with acids, DEP does not recommend using this preservative.

5.3.1.2. Properly pack the samples (see FS 2004, section 5), and place all samples on wet ice.

5.3.1.3. Analyze unpreserved samples (no acid) within 48 hours.

5.3.1.4. Analyze acid-preserved samples within the specified 14-day holding time.

5.3.1.5. Analyze unpreserved samples that have been collected in a septum vial with premeasured analyte-free water within 48 hours.

5.3.1.6. If unpreserved samples collected in a septum vial with premeasured analyte-free water are frozen to -10°C at the laboratory within 48 hours of sample collection, analyze the samples within 14 days.

5.3.1.7. Analyze samples that have been collected with and transported in a sealed coring device within 48 hours.

5.3.1.8. If unpreserved samples collected in a sealed coring device are extruded from the corer into an appropriate liquid and frozen to -10°C at the laboratory within 48 hours of sample collection, analyze the samples within 14 days.

5.3.2. High Level (> 200 µg/kg volatile organics)

5.3.2.1. Properly pack the samples (see FS 2004, section 5), and place all samples on wet ice.

5.3.2.2. Analyze samples that have been collected with and transported in a sealed coring device within 48 hours.

5.3.2.3. If unpreserved samples collected in a sealed coring device are extruded from the corer into an appropriate liquid and stored at 4°C at the laboratory within 48 hours of sample collection, analyze the samples within 14 days.

5.3.2.4. Analyze samples that that have been preserved in methanol in the field within 14-days.

6. BULK SAMPLES: The collection of bulk samples will depend on the data quality objectives of the project.

6.1. Do not composite or mix VOC samples unless required by the DEP program or if mandated by a formal DEP document (permit, order or contract).

6.2. Select sampling points from which to collect each aliquot.

6.3. Using the appropriate sampling technique, collect equal aliquots (same sample size) from each location and place in a properly cleaned container.

6.3.1. **Combine the aliquots of the sample directly in the sample container with no pre-mixing**..

6.3.2. Pack soil tightly minimizing as much headspace as possible in the sample container.

6.3.3. Cap container tightly with Teflon side facing sample.

6.4. Record the amount of each aliquot (volume or weight) in the field notes.

6.5. Label container. Refer to FS 1000, Table FS 1000-7 for preservation and holding time requirements.

6.6. Notify the laboratory that the sample is an unmixed composite sample, and request that the sample be thoroughly mixed before sample preparation or analysis.

FS 3100. Surface Soil Sampling

Surface soil is generally classified as soil between the ground surface and 6-12 inches below ground surface.

1. Remove leaves, grass and surface debris from the area to be sampled.
2. Collect samples for volatile organic analyses as described in FS 3000, section 5.
3. Select an appropriate precleaned sampling device and collect the sample.
4. Transfer the sample to the appropriate sample container.
5. Clean the outside of the sample container to remove excess soil.

6. Label the sample container, place on wet ice to preserve to 4°C and complete the field notes.

FS 3200. Subsurface Soil Sampling

Interval begins at approximately 12 inches below ground surface.

FS 3210. SAMPLE COLLECTION PROCEDURE

Use the following after the desired depth has been reached by one of the methods outlined in FS 3220.

1. Collect samples for volatile organic analyses as described in FS 3000, section 5.

2. For other analyses, select an appropriate precleaned sampling device and collect the sample.

3. Transfer the sample to the appropriate sample container.

4. Clean the outside of the sample container to remove excess soil.

5. Label the sample container, place on wet ice to preserve to 4°C and complete the field notes.

FS 3220. REACHING THE APPROPRIATE DEPTH

1. SHOVELS AND DIGGERS: Used for soils from approximately 12 inches to a point when using the implement becomes impractical.

   1.1. Dig a hole or trench to the required depth.

   1.2. Follow the sample collection procedures outlined in FS 3210.

2. BACKHOE: Used for soils from approximately 12 inches to a point when using the implement becomes impractical.

   2.1. Dig a trench to the appropriate depth.

   2.2. Expose the sample, in the trench, by using a precleaned spoon, spatula or equivalent to clean away the soil that came in contact with the backhoe bucket.

   2.3. Use a **second** precleaned utensil to actually collect the sample from the trench.

   2.4. Follow the procedures outlined in FS 3210 to collect the sample.

3. BUCKET AUGERS AND HOLLOW CORERS: Suitable to reach soils from approximately 12 inches to a point when using the implement becomes impractical.

   3.1. Push and rotate the auger into the soil until the bucket is filled.

   3.2. Addition of a non-contaminating sleeve may allow an undisturbed soil sample to be obtained.

      3.2.1. The device consists of a standard auger head with a removable sleeve, which is inserted into the auger barrel. In this case it is the sleeve, which fills with soil.

      3.2.2. Remove the sleeve from the auger and cap.

   3.3. If the auger hole is prone to collapse due to low cohesion in some soils, DEP recommends inserting a temporary rigid PVC casing into the hole. The casing prevents hole collapse and minimizes cross-contamination between soil zones as the auger is advanced.

After collecting the samples, remove the temporary casing (if used) and fill the hole filled with the excavated soil.

3.4. Remove the sample from the sampler by pushing or scraping the soil with an appropriate precleaned utensil into an appropriately precleaned tray or aluminum foil.

3.5. Remove any portion of the sample that has been disturbed and discard.

3.6. Follow the sample collection procedures outlined in FS 3210.

NOTE: If a confining layer has been breached during sampling, grout the hole to land surface with Type-1 Portland cement. This requirement may be different throughout Florida; contact the local Water Management District office for local requirements.

4. SPLIT SPOON SAMPLER: Suitable for reaching soils from approximately 12 inches to depths greater than 10 feet.

4.1. A split spoon sampler, useful for sampling unconsolidated soil, consists of two half cylinders (spoons) that fit together to form a tube approximately two feet in length and two inches in diameter.

4.1.1. The cylindrical arrangement is maintained by a retaining head and bit rings that screw on at each end of the split spoon.

4.1.2. The bit ring has beveled edges to facilitate sampling as the split spoon is forced into the ground.

4.1.3. Advance the sampler using the weight of the drilling stem and rods or a mechanical hammer.

4.1.4. Insert a catcher device in the head ring to prevent loss of unconsolidated sample during recovery.

4.2. After retrieving the split spoon sampler, expose the soil by unscrewing the bit and head rings and splitting the barrel.

4.3. If the recovery is enough to accommodate discarding a portion of the sample, discard the top and bottom two to three inches of the sample.

4.4. For volatile organic compounds collect the sample immediately from the **center portion of the split spoon** using the procedures described in FS 3000, section 5.

4.5. For other analyses, slice the sample from the center portion of the split spoon using a clean, decontaminated utensil.

4.6. Select an appropriate precleaned sampling device and collect the sample.

4.7. Transfer the sample to the appropriate sample container.

4.8. Clean the outside of the sample container to remove excess soil.

4.9. Label the sample container, place on wet ice to preserve to 4°C and complete the field notes.

5. DIRECT PUSH RIGS: May be used for depths greater than 10 feet below ground surface.

5.1. Liners: The clear liners are used with direct push rigs. This method is appropriate only for unconsolidated materials. The sampling depth that can be achieved varies depending on the rig and the lithologies that are encountered. Typically, the rig operator will:

- Place the liner inside the metal probe rod
- Select a point holder with an opening appropriate for the site lithology and screw it on the probe rod
- Advance the rod a full rod length
- Retrieve the rod
- Remove the point holder
- Remove the liner, and
- Slice the liner to expose the soil.

5.2. After the liner has been sliced, follow the procedures outlined in FS 3210, collecting volatile organic samples (if needed) immediately after the liner is sliced.

5.3. If samples for organic vapor analysis screening are required, collect them by slicing the sample(s) using a clean, decontaminated utensil and place them in 8-ounce (preferred) or 16-ounce jars, immediately cover the opening with aluminum foil and screw on the lid ring. If the contamination is derived from petroleum products, it is acceptable to use a clean gloved hand to transfer the sample(s) to the sample container(s).

5.4. For other analyses, slice the sample from the center portion of the split spoon using a clean, decontaminated utensil.

5.5. Select an appropriate precleaned sampling device and collect the sample.

5.6. Transfer the sample to the appropriate sample container.

5.7. Clean the outside of the sample container to remove excess soil.

5.8. Label the sample container, place on wet ice to preserve to 4°C and complete the field notes.

6. SHELBY TUBE SAMPLER

6.1. The Shelby tube sampler is used to sample unconsolidated soil and consists of a tube approximately 30 inches long and two inches (or larger) in diameter.

6.2. One end of the tube has edges beveled into a cutting edge. The other end can be mounted to an adapter, which allows attachment to the drilling rig assembly.

6.3. After drilling to the required depth with an auger or rotary drill bit, a soil sample is obtained through the auger or directly in the borehole.

6.4. Push the Shelby tube into the soil using the drilling rig's hydraulic ram or manually with a sledge hammer.

6.5. Remove the tube from the sampler head.

6.6. Extrude the sample from the Shelby tube.

6.7. Use a decontaminated utensil to remove any portion of the sample that has been disturbed.

6.8. Collect samples for volatile organics immediately from the center portion of the Shelby tube using the procedures described in FS 3000, section 5.

6.9. For other analyses, slice the sample from the center portion of the Shelby tube using a clean, decontaminated utensil.

6.10. Transfer the sample to the appropriate sample container.

6.11. Clean the outside of the sample container to remove excess soil.

6.12. Label the sample container, place on wet ice to preserve to 4°C and complete the field notes.

7. CORE BARREL

7.1. A standard core barrel is utilized when consolidated samples (such as limestone or dolomite) are to be sampled.

7.1.1. The core barrel is a cylinder approximately three feet long and two inches in diameter.

7.1.2. The barrel has a removable head ring with small embedded diamonds which allow the device to cut through rock or consolidated soil as the drilling rods are rotated.

7.2. Retrieve the sample core by unscrewing the head ring and sliding the sample into a precleaned container.

7.3. Use a decontaminated utensil to remove any portion of the sample that has been disturbed.

7.4. Remove the sample from the sampler (corer) with a precleaned tool.

7.5. Transfer the sample to the appropriate sample container.

7.6. Clean the outside of the sample container to remove excess soil.

7.7. Label the sample container, place on wet ice to preserve to 4°C and complete the field notes.

FT 1000. GENERAL FIELD TESTING AND MEASUREMENT

Use the following SOPs in conjunction with FT 1000:

- FD 1000 Documentation Procedures
- FM 1000 Field Planning and Mobilization
- FS 1000 General Sampling Procedures
- FT 1100 through FT 3000 Specific Field Testing Procedures

1. INTRODUCTION

1.1. Scope and Applicability: SOPs FT 1100 to FT 3000 outline procedures to conduct field testing measurements and observations. They include the parameters that are measured *in-situ* or in a field-collected sample. Additionally some samples with allowable extended holding times may be collected for laboratory measurement, as described in the specific FT-series SOPs. Included in SOPs FT 1100 to FT 3000 are:

- FT 1100 Field Measurement of Hydrogen Ion Activity (pH)
- FT 1200 Field Measurement of Specific Conductance (Conductivity)
- FT 1300 Field Measurement of Salinity
- FT 1400 Field Measurement of Temperature
- FT 1500 Field Measurement of Dissolved Oxygen (DO)
- FT 1600 Field Measurement of Turbidity
- FT 1700 Field Measurement of Light Penetration (Secchi Depth and Transparency)
- FT 1800 Field Measurement of Water Flow and Velocity
- FT 1900 Continuous Monitoring with Installed Meters
- FT 2000 Field Measurement of Residual Chlorine
- FT 3000 Aquatic Habitat Characterization

1.2. Exclusions: **If proposed for experimental purposes, field-screening procedures employing techniques not addressed in these SOPs** must be submitted to the DEP site or project manager. Such procedures must be addressed for each program or project dealing specifically with the planning and design of sampling events. Data quality objectives for quantitative assessment preclude the use of field-screening procedures for regulatory purposes.

1.3. Expectations and Requirements:

1.3.1. In some cases, specific instruments are identified in the SOP, with detailed instruction provided on their use. If you are using a different instrument from that identified in the SOP, follow the manufacturer's instructions for assembly, operation, and maintenance.

1.3.2. When required, the FT-series SOPs outline the instrument specifications. A field instrument must meet the stated requirements.

1.3.3. The FT-Series SOPs specify the calibration requirements for each method. Although instruments may vary in configuration or operation, the specified calibration requirements must be met.

1.3.3.1. Where applicable to the FT-series SOP, use the minimum number of calibration standards specified.

1.3.3.2. Do not establish the lower limit of the quantitative calibration bracket with "zero" solutions, quality control blanks or reagent dilution water.

1.3.4. Ensure that all equipment is in proper working condition, calibrated, and that batteries are properly charged before using the equipment for field testing measurements.

1.3.5. If reagents or standards are prepared from stock chemicals, they must be analytical reagent grade or better. Some procedures may specify a higher grade or assay of reagent or standard.

1.4. Recommendations for Use of Grab Samples or *in situ* Field Testing Measurements:

1.4.1. Use *in situ* readings where practical for field measurements in surface water and wastewater.

1.4.2. Use *in situ* readings or flow-through containers for field measurements for groundwater stabilization during purging and for other applications where groundwater monitoring measurements are required.

1.4.3. If grab samples are collected for measurement where allowed in the individual FT-series SOP, measure samples within fifteen (15) minutes of collection when immediate analysis is specified per Table FS 1000-4 and FS 1000-5. Otherwise, analyze grab samples within the applicable holding times specified in Table FS 1000-4 and FS 1000-5.

2. MINIMUM CALIBRATION REQUIREMENTS:

2.1. Calibration Definitions: This section outlines the essential calibration concepts that must be applied to each field test. Specific requirements for calibration are addressed in the individual SOPs.

2.1.1. Initial Calibration (IC): The instrument or meter electronics are adjusted (manually or automatically) to a theoretical value (e.g., dissolved oxygen saturation) or a known value of a calibration standard.

2.1.2. Initial Calibration Verification (ICV): The instrument or meter calibration is checked or verified directly following initial calibration by measuring a calibration standard of known value as if it were a sample and comparing the measured result to the calibration acceptance criteria listed in the SOP.

2.1.3. Continuing Calibration Verification (CCV): The instrument or meter calibration is checked or verified by measuring a calibration standard of known value as if it were a sample and comparing the measured result to the calibration acceptance criteria listed in the SOP.

2.1.4. Chronological Calibration Bracket: The interval of time between verifications within which environmental sample measurements must occur. The instrument or meter

is calibrated or verified before and verified after the time of environmental sample measurement(s).

2.1.5. Quantitative Calibration Bracket: The instrument or meter is calibrated or verified at two known values that encompass the range of observed environmental sample measurement(s).

2.1.6. Acceptance Criteria: The numerical limits within which calibration verifications are acceptable.

2.2. Calibration Activities: Specific calibration procedures are given in the individual SOPs.

2.2.1. Chronological Calibration Bracket:

2.2.1.1. Ensure that the field test result is preceded by an acceptable ICV or CCV and followed by an acceptable CCV.

2.2.1.2. Specific requirements for chronological bracketing are addressed in the individual FT-series SOPs.

2.2.2. Quantitative Calibration Bracket:

2.2.2.1. Choose two standards that bracket the range of sample measurements. These standards may be used for initial calibrations or for verifications.

2.2.2.2. Specific requirements for quantitative bracketing are addressed in the individual FT-series SOPs.

2.2.3. Initial Calibration: Calibrate if no initial calibration has been performed or if a calibration verification does not meet acceptance criteria. Do not reuse standards for initial calibrations.

Table FT 1000-1: Field Testing Acceptance Criteria

Parameter	Acceptance Criteria
pH (FT 1100)	**± 0.2 Standard pH Units of buffer or more stringent program criteria**
Specific Conductance (FT 1200)	**± 5% of standard value**
Temperature (FT 1400)	**± 0.2°C of NIST-traceable value (with correction factors) Verification over range of applicable values**
Dissolved Oxygen (FT 1500)	**± 0.3 mg/L of theoretical value (see Table FT 1500-1)**
Turbidity (FT 1600)	**0.1-10 NTU: ± 10% of standard value 11-40 NTU: ± 8% of standard value 41-100 NTU: ± 6.5% of standard value > 100 NTU: ± 5% of standard value**
Total Residual Chlorine (FT 2000)	**0.995 calibration curve correlation coefficient ± 10% of primary standard value ± 10% of secondary standard value Color comparator acceptance criterion: ± 10% of primary standard value**

2.2.4. Initial Calibration Verification:

2.2.4.1. Perform an ICV immediately after calibration. All ICVs must meet the calibration acceptance criteria specified in the applicable FT-series SOP. See Table FT 1000-1 for a list of acceptance criteria for the most common field testing procedures.

2.2.4.2. If an ICV fails to meet acceptance criteria, immediately recalibrate the instrument using the applicable initial calibration procedure or remove it from service.

2.2.5. Continuing Calibration Verification: Perform a CCV at no more than 24-hour intervals from previous verification, except where noted for individual FT-series SOPs.

2.2.5.1. If historically generated data demonstrate that a specific instrument remains stable for longer periods of time, the time interval between calibration verifications may be increased.

2.2.5.2. Base the selected time interval on the shortest interval that the instrument maintains stability. If CCVs consistently fail, shorten the time period between verifications or replace/repair the instrument.

2.2.5.3. All CCVs must meet the calibration acceptance criteria specified in the applicable FT-series SOP. See Table FT 1000-1 for a list of acceptance criteria for the most common field testing procedures.

2.2.5.4. If a CCV fails to meet acceptance criteria perform one or more of the following procedures as necessary:

- Reattempt the CCV again within the chronological bracket time interval without changing the instrument calibration. Do not perform maintenance, repair, or cleaning of the instrument or probe. Probes may be rinsed with analyte-free water or fresh verification standard. The CCV may be reattempted with a fresh aliquot of verification standard.
- Perform the initial calibration, perform an ICV, re-analyze the sample(s), and perform a CCV.
- Report all results between the last acceptable calibration verification and the failed calibration verification as estimated (report the value with a "J"). Include a narrative description of the problem in the field notes.

2.2.5.5. For installed instruments that are deployed for extended periods of time or used for continuous monitoring, see FT 1900.

2.2.5.6. Shorten the time period between verification checks or replace/repair the instrument.

2.2.6. Determining the Values of Secondary Standards: Use only those standards recommended by the manufacturer for a specific instrument. Only use secondary standards for continuing calibration verifications. See the individual FT-series SOPs for specific procedures for use of secondary standards. At documented intervals, determine or verify the values of secondary standards immediately after performing an initial calibration or after verifying the calibration with primary standards. Read each secondary standard as a sample. This result must be within the manufacturer's stated tolerance range and +/- 10% of the stated standard value. If the +/- 10% criterion is not

met, assign this reading as the value of the standard. If the reading is outside the manufacturer's stated tolerance range, discard the secondary standard.

2.2.7. **More frequent calibration verifications may be required for discharge permit compliance measurements or other regulatory requirements.**

3. PREVENTIVE MAINTENANCE: Record all maintenance and repair notes in the maintenance logbook for each meter (see FS 1007). If rental equipment is used, a log is not required. However, the origin (i.e., rental company), rental date, equipment type, model number, and identification number (if applicable) must be entered into the field notes or a rental equipment notebook.

4. DOCUMENTATION

4.1. Standard and Reagent Documentation: Document information about standards and reagents used for calibrations, verifications, and sample measurements.

4.1.1. Note the date of receipt, the expiration date and the date of first use for all standards and reagents.

4.1.1.1. Document acceptable verification of any standard used after its expiration date.

4.1.2. Record the concentration or other value for the standard in the appropriate measurement units.

4.1.2.1. Note vendor catalog number and description for pre-formulated solutions as well as for neat liquids and powdered standards.

4.1.2.2. Retain vendor assay specifications for standards as part of the calibration record.

4.1.3. Record the grade of standard or reagent used.

4.1.4. When formulated in-house, document all calculations used to formulate calibration standards.

4.1.4.1. Record the date of preparation for all in-house formulations.

4.1.5. Describe or cite the procedure(s) used to prepare any standards in-house (DEP SOP or internal SOP).

4.2. Field Instrument Calibration Documentation: Document acceptable calibration and calibration verification for each instrument unit and field test or analysis, linking this record with affected sample measurements.

4.2.1. Retain vendor certifications of all factory-calibrated instrumentation.

4.2.2. Designate the identity of specific instrumentation in the documentation with a unique description or code for each instrument unit used.

4.2.2.1. Record the manufacturer name, model number, and identifying number such as a serial number for each instrument unit.

4.2.3. Record the time and date of all initial calibrations and all calibration verifications.

4.2.4. Record the instrument reading (value in appropriate measurement units) of all calibration verifications.

4.2.5. Record the name of the analyst(s) performing the calibration.

4.2.6. Document the specific standards used to calibrate or verify the instrument or field test with the following information:

- Type of standard or standard name (e.g., pH buffer)
- Value of standard, including correct units (e.g., pH = 7.0 SU)
- Manufacturer's tolerance range for secondary standards
- Link to information recorded according to section 4.1 above

4.2.7. Retain manufacturers' instrument specifications.

4.2.8. Document whether successful initial calibration occurred.

4.2.9. Document whether each calibration verification passed or failed.

4.2.10. Document any corrective actions taken to correct instrument performance according to records requirements of FD 3000.

4.2.10.1. Document the date and time of any corrective actions.

4.2.10.2. Note any incidence of discontinuation of use of the instrument due to calibration failure.

4.2.11. Describe or cite the specific calibration or verification procedure performed (DEP SOP or internal SOP).

4.3. Record all field-testing measurement data, to include the following:

- Project name
- Date and time of measurement or test (including time zone, if applicable)
- Source and location of the measurement or test sample (e.g., monitoring well identification number, outfall number, station number or other description)
- Latitude and longitude of sampling source location (if required)
- Analyte or parameter measured
- Measurement or test sample value
- Reporting units
- Initials or name of analyst performing the measurement
- Unique identification of the specific instrument unit(s) used for the test(s)

Appendix FT 1000
Tables, Figures and Forms

Table FT 1000-1 Field Testing Acceptance Criteria

Table FT 1000-1: Field Testing Acceptance Criteria	
Parameter	**Acceptance Criteria**
pH (FT 1100)	**± 0.2 Standard pH Units of buffer or more stringent program criteria**
Specific Conductance (FT 1200)	**± 5% of standard value**
Temperature (FT 1400)	**± 0.2°C of NIST-traceable value (with correction factors) Verification over range of applicable values**
Dissolved Oxygen (FT 1500)	**± 0.3 mg/L of theoretical value (see Table FT 1500-1)**
Turbidity (FT 1600)	**0.1-10 NTU: ± 10% of standard value 11-40 NTU: ± 8% of standard value 41-100 NTU: ± 6.5% of standard value > 100 NTU: ± 5% of standard value**
Total Residual Chlorine (FT 2000)	**0.995 calibration curve correlation coefficient ± 10% of primary standard value ± 10% of secondary standard value Color comparator acceptance criterion: ± 10% of primary standard value**

FT 1100. Field Measurement of Hydrogen Ion Activity (pH)

Use in conjunction with:

- FT 1000 General Field Testing and Measurement
- FQ 1000 Field Quality Control Requirements
- FS 1000 General Sampling Procedures
- FD 1000 Documentation Procedures

1. Equipment and Supplies

1.1. Field Instrument: Use any pH meter consisting of a potentiometer, a glass electrode, a reference electrode, and a temperature-compensating device.

1.1.1. For routine fieldwork use a pH meter accurate and reproducible to at least 0.2-unit in the range of 0.0 to 14.0 units, and equipped with temperature-compensation adjustment. Record the pH value in pH units to one decimal place.

1.1.2. Advanced silicon chip pH sensors (with digital meters) may be used if demonstrated to yield equivalent performance to glass electrode sensors for the intended application.

1.2. Standards: Purchased or laboratory-prepared standard buffer solutions of pH values that bracket the expected sample pH range. Use buffers with nominal values of 4.0, 7.0 and 10.0 units for most situations. If the sample pH is outside the range of 4.0 to 10.0, then use two buffers that bracket the expected range with the pH 7 buffer being one of the two buffers. Alternatively, prepare appropriate standards per table I in method SM4500-H^+-B.

1.3. Recordkeeping and Documentation Supplies:

- Field notebook (w/ waterproof paper is recommended) or forms
- Indelible pens

2. Calibration and Use

2.1. General Concerns

2.1.1. The acceptance criterion for the initial calibration or the calibration verification is a reading of the standard within +/- 0.2-unit of the expected value.

2.1.2. On a weekly basis, check the calibration to ensure the % theoretical slope is greater than 90% (if applicable to your instrument type).

2.1.2.1. Note the % slope in the calibration records.

2.1.2.2. A % slope of less than 90% indicates a bad electrode that must be changed or repaired.

2.1.2.3. If % slope cannot be determined on your meter, or the manufacturer's optimum specifications are different, follow the manufacturer's recommendation for maintaining optimum meter performance.

2.2. Interferences

2.2.1. Sodium at pH $\geq$ 10.0 units can be reduced or eliminated by using a low sodium error electrode.

2.2.2. Coatings of oils, greases, and particles may impair the electrode's response. Pat the electrode bulb dry with lint-free paper or cloth and rinse with de-ionized water. For cleaning hard-to-remove films, use acetone very sparingly so that the electronic surface is not damaged.

2.2.3. Temperature effects on the electrometric measurement of pH are controlled by using instruments having temperature compensation or by calibrating the meter at the temperature of the samples.

2.2.4. Poorly buffered solutions with low specific conductance (< 200 μmhos/cm) may cause fluctuations in the pH readings. Equilibrate electrode by immersing in several aliquots of sample before taking pH.

2.2.5. Ensure stable sample and sensor temperature before calibrating or taking sample readings. Drifting sensor or sample temperature may produce erroneous sample measurements, calibrations, or verifications.

2.2.6. Thoroughly rinse the pH sensor with deionized water or fresh buffer standard when calibrating or verifying the calibration or when taking sample measurements. For in-situ measurements, ensure adequate flushing of the sensor with fresh sample water prior to taking measurements. Any residual standard, sample or deionized water remaining on the sensor may affect the measurement of the subsequent standard or sample. This is especially true when samples or standards of widely different pH value are successively measured.

2.2.7. Drifting readings or an inability to calibrate the sensor may also indicate a fouled electrode. Clean the electrode per the manufacturer's instructions or replace.

2.3. Calibration: Follow the manufacturer's calibration instructions specific to your meter. Most instruments allow for a two-point calibration and a few models can perform a three-point calibration. Use the appropriate number of standard buffer solutions for calibration. Do not reuse buffers for initial calibrations.

2.3.1. Rinse the probe with de-ionized water (DI) before and between each standard buffer solution.

2.3.2. Follow the calibration activities specified in FT 1000, section 2.2.

2.3.2.1. Perform an initial calibration using at least two buffers. Always use a pH 7 buffer first.

2.3.2.2. If the pH sample range is expected to be wider than the range established by a two-point calibration (e.g., some samples at pH 4 and others at pH 8), then add a third calibration point. If the instrument cannot be calibrated with three buffers, the third buffer may be used as the initial calibration verification to extend the range.

2.3.2.3. After initial calibration, immediately perform an initial calibration verification (ICV). Read a buffer as a sample. To be acceptable, a calibration verification must be within +/- 0.2 pH units of the stated buffer value. For example, if reading the pH 4.0 buffer, the result must be in the 3.8 to 4.2 range. Certain regulatory programs may have more stringent acceptance criteria.

2.3.2.4. After sample measurement(s), perform a continuing calibration verification (CCV). Read a buffer as a sample. To be acceptable, a

calibration verification must be within +/- 0.2 pH units of the stated buffer value. This CCV (if within acceptance criteria) can be used as the beginning of the chronological bracket. Certain regulatory programs may have more stringent acceptance criteria.

2.4. Measuring pH *in situ*: After calibrating the multi-probe sensors as outlined in 2.3 above, follow the meter's instructions to select the display for reading the pH of the sample. Immerse the probe at the desired depth in the water and wait for stabilization of the reading before recording the measurement.

2.5. Measuring pH in Flow-through Cells: When using a flow-through cell, the procedure described above in section 2.4 is applicable.

2.6. Measuring pH in Samples: After an acceptable initial calibration or calibration verification, follow these procedures to take a pH reading of a freshly collected sample (within 15 minutes of collection).

2.6.1. Pour enough of the fresh sample into a clean cup to take the reading.

2.6.2. Place the pH electrode in the sample (in the cup) and swirl the electrode.

2.6.3. Wait for stabilization, and read the pH value.

2.6.4. Turn the meter off after the last sample reading, rinse the electrode thoroughly with de-ionized water and replace the electrode's cap.

3. PREVENTIVE MAINTENANCE: Refer to FT 1000, section 3.

4. DOCUMENTATION

4.1. Standard and Reagent Documentation: Document information about standards and reagents used for calibrations, verifications, and sample measurements.

4.1.1. Note the date of receipt, the expiration date and the date of first use for all standards and reagents.

4.1.1.1. Document acceptable verification of any standard used after its expiration date.

4.1.2. Record the concentration or other value for the standard in the appropriate measurement units.

4.1.2.1. Note vendor catalog number and description for preformulated solutions as well as for neat liquids and powdered standards.

4.1.2.2. Retain vendor assay specifications for standards as part of the calibration record.

4.1.3. Record the grade of standard or reagent used.

4.1.4. When formulated in-house, document all calculations used to formulate calibration standards.

4.1.4.1. Record the date of preparation for all in-house formulations.

4.1.5. Describe or cite the procedure(s) used to prepare any standards in-house (DEP SOP or internal SOP).

4.2. Field Instrument Calibration Documentation: Document acceptable calibration and calibration verification for each instrument unit and field test or analysis, linking this record with affected sample measurements.

4.2.1. Retain vendor certifications of all factory-calibrated instrumentation.

4.2.2. Designate the identity of specific instrumentation in the documentation with a unique description or code for each instrument unit used.

4.2.2.1. Record manufacturer name, model number, and identifying number such as a serial number for each instrument unit.

4.2.3. Record the time and date of all initial calibrations and all calibration verifications.

4.2.4. Record the instrument reading (value in appropriate measurement units) of all calibration verifications.

4.2.5. Record the name of the analyst(s) performing the calibration.

4.2.6. Document the specific standards used to calibrate or verify the instrument or field test with the following information:

- Type of standard or standard name (e.g., pH buffer)
- Value of standard, including correct units (e.g., pH = 7.0 SU)
- Link to information recorded according to section 4.1 above

4.2.7. Retain manufacturers' instrument specifications.

4.2.8. Document whether successful initial calibration occurred.

4.2.9. Document whether each calibration verification passed or failed.

4.2.10. Document any corrective actions taken to correct instrument performance according to records requirements of FD 3000.

4.2.10.1. Document date and time of any corrective action.

4.2.10.2. Note any incidence of discontinuation of use of the instrument due to calibration failure.

4.2.11. Describe or cite the specific calibration or verification procedure performed (DEP SOP or internal SOP).

4.3. Record all field-testing measurement data, to include the following:

- Project name
- Date and time of measurement or test (including time zone, if applicable)
- Source and location of the measurement or test sample (e.g., monitoring well identification number, outfall number, station number or other description)
- Latitude and longitude of sampling source location (if required)
- Analyte or parameter measured
- Measurement or test sample value
- Reporting units
- Initials or name of analyst performing the measurement
- Unique identification of the specific instrument unit(s) used for the test(s)

FT 1200. Field Measurement of Specific Conductance (Conductivity)

Use in conjunction with:

- FT 1000 General Field Testing and Measurement
- FQ 1000 Field Quality Control Requirements
- FS 1000 General Sampling
- FD 1000 Documentation Procedures

1. INTRODUCTION: Specific conductance is a useful method to approximate the total amount of inorganic dissolved solids.

1.1. Conductivity varies with temperature. For example, the conductivity of salt water increases 3%/degree C at 0°C, and only 2%/degree C at 25°C.

1.2. Record the sample temperature or adjust the temperature of the samples prior to measuring specific conductance if the conductivity instrument does not employ automatic temperature compensation and correction of the instrument display value.

2. EQUIPMENT AND SUPPLIES

2.1. Field Instrument: Any self-contained conductivity instrument suitable for field work, accurate and reproducible to 5% or better over the operational range of the instrument, and preferably equipped with temperature-compensation adjustment. See references in FT 1210 below for additional information about instruments.

2.2. Standards: Purchased or laboratory-prepared standard potassium chloride (KCl) solutions with conductivity values that bracket the expected samples' range. In the laboratory, prepare standards of appropriate conductivities per SM2510 (Conductivity, in *Standard Methods for the Examination of Water and Wastewater, American Public Health Association*). Do not reuse standards for initial calibrations.

2.3. Recordkeeping and Documentation Supplies:

- Field notebook (w/ waterproof paper is recommended) or forms
- Indelible pens

3. CALIBRATION AND USE

3.1. General Concerns

3.1.1. Follow the instrument manufacturer's instructions for the details of operating the instrument.

3.1.2. For instruments without automatic temperature compensation, attempt to adjust the temperature of the samples to 25°C. If the temperature cannot be adjusted, measure the temperature with a calibrated device (see FT 1400), record the temperature, correct for temperature (per section 3.4 below) and report the results corrected to 25°C. See references in FT 1210 below for further information about temperature correction.

3.1.3. Ensure stable sample and sensor temperature before calibrating or taking sample readings. Drifting sensor or sample temperature may produce erroneous sample measurements, calibrations or verifications.

3.1.4. Thoroughly rinse the conductivity sensor with deionized water and fresh standard when calibrating or verifying the calibration or when taking sample measurements. For in-situ measurements, ensure adequate flushing of the sensor with fresh sample water prior to taking measurements. Any residual standard, sample or deionized water remaining on the sensor may affect the measurement of the subsequent standard or sample. This is especially true when samples or low-concentration standards are measured subsequent to measuring high-concentration standards.

3.1.5. Drifting readings or an inability to calibrate the sensor may also indicate a fouled electrode. Clean the electrodes per the manufacturer's instructions.

3.1.6. When successful calibration and verification cannot be achieved after ensuring that temperatures have stabilized and the sensor electrodes are clean and free of residual sample or standard from the previous measurement, suspect opened containers of standards, especially after repeated openings, when near the manufacturer's expiration date or when little standard volume remains in the container. Low-concentration conductivity standards are seldom stable for an extended period after opening.

3.2. Calibration and Calibration Verification:

3.2.1. Follow the calibration activities specified in FT 1000, section 2.2.

3.2.2. Initial Calibration: Calibrate the meter prior to use according to the following steps:

3.2.2.1. **Do not "zero" in the meter using analyte-free water or air.**

3.2.2.2. When the sample measurements are expected to be 100 μmhos/cm or greater, use two standard potassium chloride solutions that bracket the range of expected sample conductivities. A single standard at 100 μmhos/cm standard potassium chloride solution is acceptable for situations in which all sample measurements are expected to be less than 100 μmhos/cm.

3.2.2.3. Calibrate the instrument with one of the two standards to create an upper or lower boundary for the quantitative bracket.

3.2.2.4. Verify the calibration of the instrument with the second standard, quantitatively bracketing the range of expected sample values.

3.2.2.5. If the instrument can be calibrated with more than one standard, choose additional calibration standards within the range of expected sample values. The second standard in section 3.2.2.3 above may be used as an additional calibration standard.

3.2.2.6. Note: If all samples are expected to be less than 100 μmhos/cm, only one standard at 100 μmhos/cm standard potassium chloride solution is required.

3.2.3. Acceptability: Accept the calibration if the meter reads within +/- 5% of the value of any calibration standard used to verify the calibration. For example, the acceptance range for a 100 μmhos/cm standard is 95 to 105 μmhos/cm. If the meter does not read within +/- 5% of each calibration verification standard, determine the cause of the problem and correct before proceeding.

3.2.4. Temperature Correction: Most field instruments read conductivity directly. If the meter does not automatically correct values to 25°C, calculate correction factors using

the procedure in section 3.4 below. Record all readings and calculations in the calibration records.

3.2.5. Continuing Calibration Verification: Check the meter in read mode with at least one KCl standard with a specific conductance which quantitatively brackets the conductivity measured in environmental samples. The reading for the calibration verification must also be within +/- 5% of the standard value (see 3.2.3 above).

3.2.5.1. If new environmental samples are encountered outside the range of the initial calibration in 3.2.2 above, verify the instrument calibration with an additional standard that brackets the range of new sample values. If these calibration verifications fail, recalibrate the instrument as in 3.2.2.

3.2.5.2. **More frequent calibration verifications may be required for discharge permit compliance measurements or other regulatory requirements.**

3.3. Measuring Specific Conductance of Samples:

3.3.1. Follow manufacturer's instructions for sample measurement.

3.3.2. Immerse or place the conductivity probe or sensor in situ at a measuring location representative of the sampling source.

3.3.3. Allow the conductivity instrument to stabilize.

3.3.4. Measure the water temperature (if necessary for manual temperature compensation) and record the temperature. See FT 1400 for temperature measurement procedures.

3.3.5. If the meter is equipped with manual temperature compensation, adjust the conductivity meter to the water temperature per manufacturer's instructions.

3.3.6. If the conductivity meter has a set of positions that multiply the reading by powers of ten in order to measure the full range of potential conductivities, set this dial to the correct range in order to take a reading.

3.3.7. Record the sample conductivity measurement reading within 15 minutes of water sample collection.

3.3.8. Rinse off the probe with de-ionized water. Follow manufacturer's instructions for probe storage between use.

3.4 Calculations for Temperature Compensation

If the meter does not automatically correct for temperature (manual or automatic adjustment), or if a probe with a cell constant other than 1 is used, the following formula must be used to normalize the data to 25°C:

$$K = \frac{(K_m)(C)}{1 + 0.0191(T-25)}.$$

Where: K = conductivity in μmhos/cm at 25°C

K_m = measured conductivity in μmhos/cm at T degrees C

C = cell constant

T = measured temperature of the sample in degrees C

If the cell constant is 1, the formula for determining conductivity becomes:

$$K = \frac{(K_m)}{1 + 0.0191(T-25)}.$$

Refer to SM2510B, 20th edition, if other calculations (i.e., determining cell constant, etc.) are required. See FT 1210 below.

3.5 *In situ* Measurements at Depth or With Flow-through Cells: After calibrating the instrument as outlined in 3.2 above, follow the manufacturer's instructions to measure the conductivity of the sample.

3.5.1. For *in situ* measurements immerse the probe at the desired depth and wait for stabilization of the reading and record its value. Follow a similar procedure when using a flow-through cell.

3.5.1.1 Preferably measure groundwater sample conductivity *in situ* with a downhole probe or in a flow-through system.

4. PREVENTATIVE MAINTENANCE: Refer to FT 1000, section 3.

5. DOCUMENTATION

5.1. Standard and Reagent Documentation: Document information about standards and reagents used for calibrations, verifications and sample measurements.

5.1.1. Note the date of receipt, the expiration date and the date of first use for all standards and reagents.

5.1.1.1. Document acceptable verification of any standard used after its expiration date.

5.1.2. Record the concentration or other value for the standard in the appropriate measurement units.

5.1.2.1. Note vendor catalog number and description for preformulated solutions as well as for neat liquids and powdered standards.

5.1.2.2. Retain vendor assay specifications for standards as part of the calibration record.

5.1.3. Record the grade of standard or reagent used.

5.1.4. When formulated in-house, document all calculations used to formulate calibration standards.

5.1.4.1. Record the date of preparation for all in-house formulations.

5.1.5. Describe or cite the procedure(s) used to prepare any standards in-house (DEP SOP or internal SOP).

5.2. Field Instrument Calibration Documentation: Document acceptable calibration and calibration verification for each instrument unit and field test or analysis, linking this record with affected sample measurements.

5.2.1. Retain vendor certifications of all factory-calibrated instrumentation.

5.2.2. Designate the identity of specific instrumentation in the documentation with a unique description or code for each instrument unit used.

5.2.2.1. Record manufacturer name, model number, and identifying number such as a serial number for each instrument unit.

5.2.3. Record the time and date of all initial calibrations and all calibration verifications.

5.2.4. Record the instrument reading (value in appropriate measurement units) of all calibration verifications.

5.2.5. Record the name of the analyst(s) performing the calibration.

5.2.6. Document the specific standards used to calibrate or verify the instrument or field test with the following information:

- Type of standard or standard name (e.g., conductivity standard)
- Value of standard, including correct units (e.g., conductivity = 100 µmhos/cm)
- Link to information recorded according to section 5.1 above

5.2.7. Retain manufacturers' instrument specifications.

5.2.8. Document whether successful initial calibration occurred.

5.2.9. Document whether each calibration verification passed or failed.

5.2.10. Document any corrective actions taken to correct instrument performance according to records requirements of FD 3000.

5.2.10.1. Document date and time of any corrective action.

5.2.10.2. Note any incidence of discontinuation of use of the instrument due to calibration failure.

5.2.11. Describe or cite the specific calibration or verification procedure performed (DEP SOP or internal SOP).

5.3. Record all field-testing measurement data, to include the following:

- Project name
- Date and time of measurement or test (including time zone, if applicable)
- Source and location of the measurement or test sample (e.g., monitoring well identification number, outfall number, station number or other description)
- Latitude and longitude of sampling source location (if required)
- Analyte or parameter measured
- Measurement or test sample value
- Reporting units
- Initials or name of analyst performing the measurement
- Unique identification of the specific instrument unit(s) used for the test(s)

FT 1300. Field Measurement of Salinity

Use in conjunction with:

- FT 1000 General Field Testing and Measurement
- FQ 1000 Field Quality Control Requirements
- FS 1000 General Sampling Procedures
- FD 1000 Documentation Procedures

1. INTRODUCTION: Salinity is an important property of industrial and natural waters. This field parameter is also important for assessing the source or origin of effluents and of the mixing between fresh and marine waters in coastal regions, in both surface water and groundwater.

1.1. Salinity is a unit-less parameter since by definition it is the ratio of the mass of dissolved salts to the total mass of a given volume of water. Thus, salinity values are commonly expressed as "grams of salt/kilograms of water" or $^{o}/_{oo}$.

1.2. Salinity is determined by using indirect methods involving the measurement of a related physical property such as conductivity, density, sound speed, or refractive index. The commonly used procedures in the field are determination of conductivity or density of the sample.

1.3. The sample salinity is calculated from an empirical relationship between salinity and the physical property as determined from a standard solution. Refer to the referenced method SM2520 for further discussions on these topics.

1.4. Because of its high sensitivity and easy of measurement, the conductivity method is most often used to determine the salinity. (Note – using a hydrometer to measure the density or the specific gravity to obtain an approximate salinity value is not recommended for reporting purposes.)

2. EQUIPMENT AND SUPPLIES

2.1. Field Instrument: Depending on the chosen method, use:

2.1.1. Any self-contained conductivity instrument with a platinum or graphite electrode type cell, and a temperature sensor. Some conductivity instruments have meter scales pre-calibrated for salinity and are sometimes referred to as Salinometers. For routine fieldwork use a conductivity meter accurate and reproducible to at least 5% or 1 μmho/cm (whichever is greater), and equipped with temperature-compensation adjustment; or

2.1.2. A precision "vibrating flow densimeter" (see Millero & Poisson, 1981) and a field thermometer.

2.2. Standards:

2.2.1. Purchased or laboratory-prepared Standard Seawater and/or potassium chloride (KCl) standards of appropriate equivalent salinities.

2.2.1.1. In the laboratory, prepare the Standard Seawater per recipe in method SM2520 and SM8010 (Table III), and standard KCl solutions per recipe in method SM2510 (American Public Health Association, American Water Works Association, Water Pollution Control Federation, *Standard Methods for the Examination of Water and Wastewater)*.

2.2.2. De-ionized water for calibration of the densimeter (if used).

2.3. Recordkeeping and Documentation Supplies:

- Field logbook (w/ waterproof paper is recommended) or field forms
- Indelible pens

3. CALIBRATION AND USE

3.1. Conductivity Method

3.1.1. Calibration: - Calibrate the instrument per manufacturer's instructions using one calibration standard, either standard seawater or a KCl solution, as applicable. The acceptance criterion for initial calibration or a calibration verification is that the instrument reading is within +/- 5% of the standard value. For example, when calibrating with standard seawater, S = 35, the meter must read in the 34 to 36 range in order to be acceptable.

3.1.1.1. Use standard seawater (S = 35) when measuring salinity in the open ocean or estuaries with a predominance of seawater.

3.1.1.2. KCl may be used in estuarine waters with low salinity (S = 0 – 40).

3.1.1.3. If verifying or calibrating with a "zero" standard, do not use analyte-free water or air check (dry electrode) as the blank.

3.1.1.4. If the meter does not provide a direct reading of salinity, use the equation found in SM2520B to convert the readings to salinity.

3.1.1.5. Follow the calibration activities in FT 1000, section 2.2.

3.1.1.6. Do not reuse standards for initial calibrations.

3.1.2. Field Use: - Rinse the probe with DI water after calibration and before each sample measurements. Follow the manufacturer's instructions for temperature compensation, if needed. Report salinities with only one decimal figure.

3.1.3. General Concerns for Conductivity Method

3.1.3.1. Ensure stable sample and sensor temperature before calibrating or taking sample readings. Drifting sensor or sample temperature may produce erroneous sample measurements, calibrations, or verifications.

3.1.3.2. Thoroughly rinse the conductivity (salinity) sensor with deionized water and fresh standard when calibrating or verifying the calibration or when taking sample measurements. For in-situ measurements, ensure adequate flushing of the sensor with fresh sample water prior to taking measurements. Any residual standard, sample, or deionized water remaining on the sensor may affect the measurement of the subsequent standard or sample. This is especially true when samples or low-concentration standards are measured subsequent to measuring high-concentration standards.

3.1.3.3. Drifting readings or an inability to calibrate the sensor may also indicate a fouled electrode. Clean the electrodes per the manufacturer's instructions.

3.1.3.4. When successful calibration and verification cannot be achieved after ensuring that temperatures have stabilized and the sensor electrodes are clean and free of residual sample or standard from the previous measurement, suspect opened containers of standards, especially after repeated openings, when near the

manufacturer's expiration date or when little standard volume remains in the container. Low-concentration conductivity standards are seldom stable for an extended period after opening.

3.2. Density Method

The vibrating flow densimeter is an instrument that allows for precise and rapid measurements of the density of a liquid, such as water. The principle of operation is the effect of the density of the sample on the frequency of a vibrating tube encased in a constant-temperature jacket. The measurement is made by passing the water (sample) through the vibrating tube and reading the period of vibration that is electronically sensed and displayed by the densimeter. The sample density (D) is proportional to the square of the period of vibration (T):

$$D = a + bT^2$$

Where a and b are terms determined by calibration, b being determined by calibration of the densimeter with Standard Seawater. The difference between the density of the sample (D) and that of pure water (D_0) is given by:

$$D - D_0 = b\,(T^2 - T_0^2)$$

Where T and T_0 are, respectively, the periods of the sample and that of pure (de-ionized) water. Using this second equation, you only have to deal with the term b for calibration purposes. Hence, the system can be calibrated with two liquids: pure water and Standard Seawater. Follow the manufacturer's instruction for calibration of the densimeter.

The salinity of the sample is determined by the one-atmosphere international equation of state for seawater. This equation relates the difference ($D - D_0$) to the practical salinity as a function of the temperature of the sample (which is also measured by the densimeter or the field thermometer). For further details on this calculation read the referenced method SM2520C.

4. PREVENTIVE MAINTENANCE: Refer to FT 1000, section 3.

5. DOCUMENTATION

5.1. Standard and Reagent Documentation: Document information about standards and reagents used for calibrations, verifications, and sample measurements.

5.1.1. Note the date of receipt, the expiration date and the date of first use for all standards and reagents.

5.1.1.1. Document acceptable verification of any standard used after its expiration date.

5.1.2. Record the concentration or other value for the standard in the appropriate measurement units.

5.1.2.1. Note vendor catalog number and description for preformulated solutions as well as for neat liquids and powdered standards.

5.1.2.2. Retain vendor assay specifications for standards as part of the calibration record.

5.1.3. Record the grade of standard or reagent used.

5.1.4. When formulated in-house, document all calculations used to formulate calibration standards.

5.1.4.1. Record the date of preparation for all in-house formulations.

5.1.5. Describe or cite the procedure(s) used to prepare any standards in-house (DEP SOP or internal SOP).

5.2. Field Instrument Calibration Documentation: Document acceptable calibration and calibration verification for each instrument unit and field test or analysis, linking this record with affected sample measurements.

5.2.1. Retain vendor certifications of all factory-calibrated instrumentation.

5.2.2. Designate the identity of specific instrumentation in the documentation with a unique description or code for each instrument unit used.

5.2.2.1. Record manufacturer name, model number, and identifying number such as a serial number for each instrument unit.

5.2.3. Record the time and date of all initial calibrations and all calibration verifications.

5.2.4. Record the instrument reading (value in appropriate measurement units) of all calibration verifications.

5.2.5. Record the name of the analyst(s) performing the calibration.

5.2.6. Document the specific standards used to calibrate or verify the instrument or field test with the following information:

- Type of standard or standard name (e.g., salinity standard)
- Value of standard, including correct units (e.g., salinity = 20 $^{o}/_{oo}$)
- Link to information recorded according to section 5.1 above

5.2.7. Retain manufacturers' instrument specifications.

5.2.8. Document whether successful initial calibration occurred.

5.2.9. Document whether each calibration verification passed or failed.

5.2.10. Document any corrective actions taken to correct instrument performance according to records requirements of FD 3000.

5.2.10.1. Document date and time of any corrective action.

5.2.10.2. Note any incidence of discontinuation of use of the instrument due to calibration failure.

5.2.11. Describe or cite the specific calibration or verification procedure performed (DEP SOP or internal SOP).

5.3. Record all field-testing measurement data, to include the following:

- Project name
- Date and time of measurement or test (including time zone, if applicable)
- Source and location of the measurement or test sample (e.g., monitoring well identification number, outfall number, station number or other description)
- Latitude and longitude of sampling source location (if required)

- Analyte or parameter measured
- Measurement or test sample value
- Reporting units
- Initials or name of analyst performing the measurement
- Unique identification of the specific instrument unit(s) used for the test(s)

FT 1400. Field Measurement of Temperature

The use of this SOP is not required when using field temperature measurement devices to monitor groundwater stabilization during the purging of groundwater monitoring wells. Field temperature measurement devices used for temperature compensation (correction) for other measurements such as dissolved oxygen, specific conductance or pH are also exempted from the requirements of this SOP. FT 1400 must be used for all other field temperature measurements required by DEP.

Use this SOP in conjunction with the following DEP SOPs:

- FT 1000 General Field Testing and Measurement
- FQ 1000 Field Quality Control Requirements
- FS 1000 General Sampling Procedures
- FD 1000 Documentation Procedures

1. EQUIPMENT AND SUPPLIES

1.1. Field Instruments: Use any of the following instrument types for performing field measurements:

- Digital thermistor (thermocouple type) and meter typical of field instruments
- Glass bulb, mercury-filled thermometer (not recommended for field ruggedness)
- Glass bulb, alcohol-filled thermometer with protective case
- Bi-metal strip/dial-type thermometer
- Advanced silicon chip temperature sensor and digital meter

1.1.1. Field instruments must be capable of measuring temperature in 0.1^{o}C increments.

1.2. Standard Thermometer: NIST-traceable Celsius certified thermometer with scale marks for every 0.1^{o}C increment, a range of 0^{o}C to 100^{o}C (or a range bracketing expected sample temperatures) and correction chart supplied with certification. The standard thermometer must have a valid certification for the period of measurement.

1.3. Recordkeeping and Documentation Supplies:

- Field notebook or forms \
- Indelible pens

2. CALIBRATION AND USE

2.1. General Concerns

2.1.1. Select a temperature measuring device meeting the requirements of section 1.1 above.

2.1.2. Dial-type and thermocouple-type devices with meters are preferred over the glass thermometers for fieldwork because of their durability and ease of reading.

2.1.2.1. Transport glass thermometers in protective cases.

2.1.2.2. Inspect glass thermometers for liquid separation. Do not use a thermometer if the liquid has separated.

2.1.2.3. Most instruments with digital display will provide more decimal figures than are significant. Record the temperature reading with only one rounded decimal figure (e.g., 25.9 instead of 25.86°C).

2.2. Calibration

2.2.1. Follow the calibration activities specified in FT 1000, section 2.2.

2.2.2. Verify all thermistor (meter) devices and field thermometers against the NIST-traceable standard thermometer at several temperatures in the expected sample measurement range, using any correction factor indicated by the certificate supplied with the NIST-traceable thermometer.

2.2.2.1. See the US Geological Survey, National Field Manual for the Collection of Water-Quality Data, Book 9, Chapter A6, Field Measurements, Section 6.1, Temperature, Techniques of Water-Resources Investigations, 4/98 for additional guidance about making temperature comparisons with the standard thermometer.

2.2.2.2. Make note of the calibration in the calibration records. See section 4 below.

2.2.2.3. The field measurement device may be used with a linear correction factor provided that the observed temperature difference with the standard thermometer is documented at incremental temperatures over the range of expected sample temperatures.

2.2.2.4. Use the resulting correction factor when making temperature measurements of samples with the field measurement device.

2.2.2.5. Prominently display the correction factor on the field measurement device, with the date last verified. A calibration correction curve or plot may also be used.

2.2.2.6 To be acceptable, a calibration verification must be within +/- 0.5°C of the corrected reading of the NIST-traceable thermometer.

2.2.2.7 Properly dispose of glass-bulb thermometers that do not meet the above calibration acceptance criteria.

2.2.3. Continuing Calibration Verifications:

2.2.3.1. Determine the maximum time between continuing calibration verifications for the specific field temperature measurement device based on instrument stability.

2.2.3.2. Verify the field measurement device against the standard NIST-traceable thermometer as in section 2.2.2 above.

2.2.4. Refer to additional calibration requirements in FT 1000, section 2.2.

2.2.5. **More frequent calibration verifications may be required for discharge permit compliance measurements or other regulatory requirements.**

2.3. Measuring Sample Temperature

2.3.1. Insert or place the thermometer or sensor *in situ* at a measuring location representative of the sampling source.

2.3.2. Allow the thermometer or temperature sensor to equilibrate to ambient *in situ* temperature.

2.3.2.1. Groundwater samples must be measured *in situ* with a downhole probe or in a flow-through container. Do not measure bailed or pumped samples in an intermediate container containing static sample.

2.3.3. Record the temperature to the nearest 0.1°C after the reading stabilizes and remains constant.

3. PREVENTIVE MAINTENANCE: Refer to FT 1000, section 3.

4. DOCUMENTATION

4.1. Standards Documentation: Document information about the NIST-traceable standard thermometer in the calibration record, including:

- Unique identification for the thermometer
- Vendor certificate of calibration, including any correction factor
- Vendor's expiration date for the certificate of calibration

4.2. Field Instrument Calibration Documentation: Document acceptable calibration and calibration verification for each instrument unit and field test or analysis, linking this record with affected sample measurements.

4.2.1. Retain vendor certifications of all factory-calibrated instrumentation.

4.2.2. Designate the identity of specific instrumentation in the documentation with a unique description or code for each instrument unit used.

4.2.2.1. Record manufacturer name, model number, and identifying number such as a serial number for each instrument unit.

4.2.3. Record the time and date of all initial calibrations and all calibration verifications.

4.2.4. Record the instrument reading (value in appropriate measurement units) of all calibration verifications.

4.2.5. Record the name of the analyst(s) performing the calibration.

4.2.6. Document the following information about initial calibration and calibration verifications and link to information recorded according to section 4.1 above:

- Details of the method used to compare the field measurement device to the NIST-traceable standard thermometer.
- Results of each calibration verification, including the expected reading (per the NIST-traceable standard thermometer)
- The actual reading of the field measurement device, using any established correction factors and correct units.

4.2.7. Retain manufacturers' instrument specifications.

4.2.8. Document whether successful initial calibration occurred.

4.2.9. Document whether each calibration verification passed or failed.

4.2.10. Document any corrective actions taken to correct instrument performance (such as a new correction factor) according to records requirements of FD 3000.

4.2.10.1. Document date and time of any corrective action.

4.2.10.2. Note any incidence of discontinuation of use of the instrument due to calibration failure.

4.2.11. Describe or cite the specific calibration or verification procedure performed (DEP SOP or internal SOP).

4.3. Record all field-testing measurement data, to include the following:

- Project name
- Date and time of measurement or test (including time zone, if applicable)
- Source and location of the measurement or test sample (e.g., monitoring well identification number, outfall number, station number or other description)
- Latitude and longitude of sampling source location (if required)
- Analyte or parameter measured
- Measurement or test sample value
- Reporting units
- Initials or name of analyst performing the measurement
- Unique identification of the specific instrument unit(s) used for the test(s)

FT 1500. Field Measurement of Dissolved Oxygen (DO)

Use in conjunction with:

- FT 1000 General Field Testing and Measurement
- FS 1000 General Sampling Procedures
- FD 1000 Documentation Procedures

1. EQUIPMENT AND SUPPLIES

1.1. Field Instruments

1.1.1. Membrane-type polarographic or galvanic electrode DO sensor with dedicated meter or configured with multi-parameter sonde

1.1.2. Luminescence-based DO sensor with dedicated meter or configured with multi-parameter sonde (see American Society for Testing and Materials, *Standard Test Methods for Dissolved Oxygen in Water*, Test Method C-Luminescence-based Sensor, D 888-05).

1.1.3. Select instrument assemblies that provide minimum precision of +/- 0.2 mg DO/L and a minimum accuracy of +/- 0.2 mg DO/L.

1.1.4. Compensate for temperature dependence of DO measurements by using instruments employing automatic temperature compensation or by manually correcting measurements in accordance with SM 4500-O G (see *Standard Methods for the Examination of Water and Wastewater*, American Public Health Association, American Water Works Association, Water Pollution Control Federation).

1.1.4.1. Calibrate on-board temperature sensors as described in FT 1400.

1.2. Standards

1.2.1. NIST-traceable Celsius thermometer with a scale marked for every 0.1^{o}C and a range of 0 to 100^{o}C.

1.2.2. Access to an organization with capability to perform the Winkler titration procedure is recommended but not mandatory.

1.2.3. A “zero-DO standard”, prepared on-site with an aliquot of the sample water, is optional. Prepare by adding excess sodium sulfite and a trace of cobalt chloride to bring the DO to zero.

1.3. Recordkeeping and Documentation Supplies:

- Field notebook (w/ waterproof paper is recommended) or forms
- Indelible pens

2. CALIBRATION AND USE: the electrode method is predominantly used *in-situ* for dissolved oxygen determinations.

2.1. General Concerns

2.1.1. Turbulence is necessary to keep a constant flow of water across the membrane-sample interface. Make sure the appropriate mechanism is working before using the probe.

2.1.2. Follow instrument manufacturer's instructions for probe storage. For example, store the probe with a cover that creates a saturated atmosphere. A cap, with a wet sponge in it, will suffice for single-parameter probes. If the sensor is in a multi-probe device, keep the protective cap chamber moist during storage.

2.1.3. Before mobilizing, check to make sure there are no bubbles beneath the probe membrane, or any wrinkles or tears in the probe membrane. If so, replace the membrane and KCL solution. Check the leads, contacts, etc. for corrosion and/or shorts if meter pointer remains off-scale, does not calibrate, or drifts.

2.1.4. Dissolved inorganic salts interfere with the performance of DO probes. For example, DO readings in salt water are affected by the salinity and must be corrected. The DO meter may adjust automatically based on readings taken from the specific conductivity/salinity probe. If corrections are not automatic the appropriate calculations must be used to correct for salinity. If automatic adjustments are used the specific conductivity/salinity probe calibration must be verified or calibrated in accordance with FT1200.

2.1.5. Reactive gases, which pass through the membrane, may interfere. For example, chlorine will depolarize the cathode and cause a high probe output. Long-term exposures to chlorine will coat the anode with the chloride of the anode metal and eventually desensitize the probe. Sulfide (from H_2S) will undergo oxidation if high enough potential (voltage) is applied, creating current flow, yielding faulty readings. If such interferences are suspected, change the membrane electrode more frequently and calibrate at more frequent intervals.

2.1.6. Ensure that the temperature of the sensor and sample are stable. Unstable temperatures will produce erroneous calibrations, verifications or sample measurements.

2.1.7. Erroneous calibrations or verifications may result if the saturated air chamber is not vented to atmospheric pressure, properly humidified and protected from temperature fluctuations produced by common field conditions such as evaporation or fluctuation in sunlight intensity.

2.2. Follow the quality control requirements for calibration (see activities in FT 1000, section 2.2).

2.3. Initial Calibration and Initial Calibration Verification

2.3.1. Air Calibration and Initial Calibration Verification (ICV): Calibrate the meter at 100% saturation. Before use, verify the meter calibration in water-saturated air to make sure it is properly calibrated and operating correctly. Make a similar verification at the end of the day or sampling event. Follow the manufacturer's instructions for your specific instrument.

2.3.1.1. Allow an appropriate warm up period before initial field calibration.

2.3.1.2. Wet the inside of the calibration chamber with water, pour out the excess water (leave a few drops), wipe any droplets off the membrane/sensor and insert the sensor into the chamber (this ensures 100% humidity).

2.3.1.3. Allow adequate time for the DO sensor and the air inside the calibration chamber to equilibrate.

2.3.1.4. Once the probe/calibration chamber is stable at ambient temperature, check the air temperature and determine, from the DO versus temperature table, what the DO saturation value should be at the observed temperature (see Table FT

1500-1, below). A stable and accurate temperature is required for a valid calibration. The acceptance criterion for DO calibration verification is +/- 0.3 mg DO/L at the observed temperature of the verification.

2.4. Continuous Calibration Verification

2.4.1. Air-Calibration Verification: DO sensor or instrument is calibrated against air that is saturated with water at a known temperature and ambient atmospheric pressure. Use Table FT 1500-1 below to verify calibration at specified temperature.

2.4.1.1. Wet the inside of the calibration chamber with water, pour out the excess water (leave a few drops) and insert the sensor into the chamber (this ensures 100-percent humidity)

2.4.1.2. Allow adequate time for the DO sensor and the air inside the calibration chamber to equilibrate.

2.4.1.3. Measure the temperature in the calibration chamber and observe the readings until the instrument stabilizes.

2.4.1.4. Use the oxygen solubility Table FT 1500-1 below to determine the DO saturation at a measured temperature and atmospheric pressure. Calculate values to the nearest tenth degree by interpolation or use an expanded version of this table found in FS 2200, which provides saturation data in 0.1 ^{o}C increments for a selected temperature range (see Table FS 2200-2).

2.4.1.5. Compare DO meter reading with value obtained from Table FT 1500-1 below to verify continuous calibration.

2.5. Additional Verifications: The following methods may be used as additional checks to verify calibration. These additional checks may be required as part of a specific permit.

2.5.1. Winkler method: This check is useful to assess the condition of the DO sensor (i.e., its degradation with time/use) and that the instrument can still maintain a valid calibration (see SM 4500-O C).

2.5.1.1. **Perform the Winkler method when required by permit or other regulation at the required calendar frequency.**

2.5.1.2. For an accuracy calibration verification using the Winkler method, follow SM 4500-O C.

2.5.1.3. Fill a clean bucket with uncontaminated or de-ionized water and place the probe into the bucket (with stirrer or equivalent mechanism turned off). Fill at least two biological oxygen demand (BOD) bottles without entraining atmospheric oxygen into the bottles. Carefully submerge the bottom of the bottle (one at a time) into the water and allow the water to fill the bottle. Place the bottle on the bottom of the bucket and carefully place stopper into it without adding atmospheric oxygen. Retrieve the bottles and determine their DO by the Winkler method (see SM4500-O-C for more details). Turn the stirrer or equivalent mechanism on and read the DO of the water in the bucket.

2.5.1.4. Adjust the DO meter according to manufacturer's instructions. Be sure to adjust the meter to the temperature of water in the bucket, and then calibrate the DO meter to read the average DO concentration of the two samples determined by the Winkler test.

2.5.2. Zero-DO Verification: The air calibration and the interfering effects of the sample can be further checked in the field by means of a "zero-DO standard"(SM 4500-O G).

2.5.2.1. Prepare this standard on-site with an aliquot of the sample by adding excess sodium sulfite and a trace of cobalt chloride to bring the DO to zero. Prepare this zero-DO standard in a beaker or a large-mouth sample container of appropriate size to insert the DO probe.

2.5.2.2. After adding the chemicals, gently swirl the water and let it sit for about 30 seconds before inserting the probe.

2.5.2.3. Read the DO of the sample. If the reading is outside the acceptance interval, the instrument must be recalibrated and/or zero-adjusted if the meter allows for this adjustment.

2.5.3. Air-Saturated Water: The DO sensor or instrument system is calibrated against water that is saturated with oxygen at a known temperature and ambient atmospheric pressure.

2.5.3.1. The temperature and conductivity of water used for calibration should be about the same as the temperature and conductivity of the water to be measured.

2.5.3.2. Place DO sensor and calibration water in a large beaker or open-mouth container.

2.5.3.3. Aerate the water for an adequate amount of time.

2.5.3.4. Determine if the water is 100 percent saturated with oxygen, and take a temperature reading. Temperature must be calibrated or verified for accuracy before DO calibration verification.

2.5.3.5. Use Table FT 1500-1 above to determine the DO saturation value at the measured water temperature. Compare DO meter reading with value obtained from Table FT 1500-1 to ensure continuous calibration.

2.6. Measuring DO in Samples:

2.6.1. Insert or place the DO probe *in situ* at a measuring location representative of the sampling source:

2.6.1.1. Take the DO of an effluent just before it enters the receiving water. If the effluent aerated prior to entering the surface water, take the DO reading in the receiving water right where it enters.

2.6.1.2. For well mixed surface waters, e.g., fast flowing streams, take the DO reading at approximately 1-2 feet below the surface or at mid-depth.

2.6.1.3. For still or sluggish surface waters, take a reading at one foot below the surface, one foot above the bottom, and at mid-depth.

2.6.1.4. If it is shallow surface waters, (less than two feet) take the reading at mid-depth.

2.6.1.5. Do not take a reading in frothy or aerated water unless required by the sampling plan.

2.6.1.6. Groundwater samples must be measured *in situ* with a downhole probe or in a flow-through container. Do not measure bailed or pumped samples in an intermediate container containing static sample.

2.6.2. Rinse probe with de-ionized water and keep the probe in the saturated atmosphere (see 2.1.2 above) between sites and events.

2.6.3. If the readings show distinct, unexplainable changes in DO levels, or when the probe has been in waters with high sulfides, recalibrate or perform maintenance per manufacturer's instructions. While taking a reading, if it is very low (e.g., below 1.0 mg/L), allow the meter to stabilize, record it and then, remove and rinse the probe, as the environment is very likely anoxic and may contain hydrogen sulfide, which can damage the probe.

2.6.4. Salinity and Temperature corrections may be necessary. Follow manufacturer instructions for automatic corrections or perform manual calculations (SM 4500-O G).

3. PREVENTIVE MAINTENANCE: Refer to FT 1000, section 3.

4. DOCUMENTATION

4.1. Standard and Reagent Documentation: Document information about standards and reagents used for verifications.

4.1.1. Note the date of receipt, the expiration date and the date of first use for all standards and reagents.

4.1.1.1. Document acceptable verification of any standard used after its expiration date.

4.1.2. Record the concentration or other value for the standard in the appropriate measurement units.

4.1.2.1. Note vendor catalog number and description for pre-formulated solutions as well as for neat liquids and powdered standards.

4.1.2.2. Retain vendor assay specifications for standards as part of the calibration record.

4.1.3. Record the grade of standard or reagent used.

4.1.4. When formulated in-house, document all calculations used to formulate calibration standards.

4.1.4.1. Record the date of preparation for all in-house formulations.

4.1.5. Describe or cite the procedure(s) used to prepare any standards in-house (DEP SOP or internal SOP).

4.2. Field Instrument Calibration Documentation: Document acceptable calibration and calibration verification for each instrument unit and field test or analysis, linking this record with affected sample measurements.

4.2.1. Retain vendor certifications of all factory-calibrated instrumentation.

4.2.2. Designate the identity of specific instrumentation in the documentation with a unique description or code for each instrument unit used.

4.2.2.1. Record the manufacturer name, model number and identifying number such as a serial number for each instrument unit.

4.2.3. Record the time and date of all initial calibrations and all calibration verifications.

4.2.4. Record the instrument reading (value in appropriate measurement units) of all calibration verifications.

4.2.5. Record the temperature associated with all calibration verifications.

4.2.6. Record the name of the analyst(s) performing the calibration.

4.2.7. Document the specific standards used to calibrate or verify the instrument or field test with the following information:

- Type of standard or standard name (e.g., saturation)
- Value of standard, including correct units (e.g., mg/L at ^{o}C)
- Link to information recorded according to section 4.1 above

4.2.8. Retain manufacturers' instrument specifications.

4.2.9. Document whether successful initial calibration occurred.

4.2.10. Document whether each calibration verification passed or failed.

4.2.11. Document any corrective actions taken to correct instrument performance according to records requirements of FD 3000.

4.2.11.1. Document the date and time of any corrective action.

4.2.11.2. Note any incidence of discontinuation of use of the instrument due to calibration failure.

4.2.12. Describe or cite the specific calibration or verification procedure performed (DEP SOP or internal SOP).

4.3. Record all field-testing measurement data, to include the following:

- Project name
- Date and time of measurement or test (including time zone, if applicable)
- Source and location of the measurement or test sample (e.g., monitoring well identification number, outfall number, station number or other description)
- Latitude and longitude of sampling source location (if required)
- Analyte or parameter measured
- Measurement or test sample value
- Reporting units
- Initials or name of analyst performing the measurement
- Unique identification of the specific instrument unit(s) used for the test(s)

Appendix FT 1500
Tables, Figures and Forms

Table FT 1500-1 Solubility of Oxygen in Water

Table FT 1500-1: Solubility of Oxygen in Water at Atmospheric Pressure[1,2]			
Temperature °C	Oxygen Solubility mg/L	Temperature °C	Oxygen Solubility mg/L
0.0	14.621	26.0	8.113
1.0	14.216	27.0	7.968
2.0	13.829	28.0	7.827
3.0	13.460	29.0	7.691
4.0	13.107	30.0	7.559
5.0	12.770	31.0	7.430
6.0	12.447	32.0	7.305
7.0	12.139	33.0	7.183
8.0	11.843	34.0	7.065
9.0	11.559	35.0	6.950
10.0	11.288	36.0	6.837
11.0	11.027	37.0	6.727
12.0	10.777	38.0	6.620
13.0	10.537	39.0	6.515
14.0	10.306	40.0	6.412
15.0	10.084	41.0	6.312
16.0	9.870	42.0	6.213
17.0	9.665	43.0	6.116
18.0	9.467	44.0	6.021
19.0	9.276	45.0	5.927
20.0	9.092	46.0	5.835
21.0	8.915	47.0	5.744
22.0	8.743	48.0	5.654
23.0	8.578	49.0	5.565
24.0	8.418	50.0	5.477
25.0	8.263		

1. The table provides three decimal places to aid interpolation

2. Under equilibrium conditions, the partial pressure of oxygen in air-saturated water is equal to that of the oxygen in water-saturated

FT 1600. Field Measurement of Turbidity

Use in conjunction with:

- FT 1000 General Field Testing and Measurement
- FS 1000 General Sampling Procedures
- FD 1000 Documentation Procedures

1. INTRODUCTION: Turbidity measures the scattering effect that suspended solids have on the propagation of light through a body of water (surface or ground waters). The higher the effect (i.e., intensity of scattered light), the higher the turbidity value. Suspended and colloidal matter such as clay, silt, finely divided organic and inorganic matter, and plankton and other microscopic organisms cause turbidity in water.

This SOP describes the use of true nephelometric measurement using instruments meeting the specifications outlined in 2.1.

Exceptions to the requirements specified in 2.1 below include:

1.1. *In situ* probes with turbidity sensors used for screening purposes (e.g., groundwater purge stabilization measurements).

1.2. Non standard light sources, detectors or other turbidity measuring devices may be proposed for use in studies that entail comparison measurements (dredge and fill) or unattended deployment for monitoring purposes.

1.3. **Do not report results from "non standard" sensors or configurations for regulatory purposes such as permit compliance unless the Department has approved the use for the specific project.**

1.4. All "non standard" instrument must be calibrated/check according to the principles outlined in this SOP.

2. EQUIPMENT AND SUPPLIES

2.1. Field Instrument: Use a turbidimeter (nephelometer) or a spectrophotometer consisting of a light source and one or more photoelectric detectors with a readout device to indicate the intensity of light. The instrument must meet these specifications:

2.1.1. The light source must have a tungsten-filament lamp operated at a color temperature between 2000 and 3000 K.

2.1.2. The distance traversed by the incident light and scattered light within the sample tube must not exceed 10 cm.

2.1.3. The light detector, positioned at 90^{o} to the incident light, must have an acceptance angle that does not exceed $\pm$ 30^{o} from 90^{o}.

2.1.4. The detector and any filter system must have a spectral peak response between 400 and 600 nanometers.

2.1.5. The instrument sensitivity must permit detection of a turbidity difference of 0.02 NTU at the 0 – 1.0 NTU scale.

2.1.6. Note: using the appropriate equipment and following the procedures in this SOP, the field accuracy of this measurement is close to %R = 100 ± 10% for turbidities in the range of 1 to 100 NTU.

2.2. Sample Cells (cuvettes): Use sample cells or tubes of clear, colorless glass or plastic.

2.2.1. Keep cells clean, both inside and out, and discard if scratched or etched.

2.2.1.1. Never handle them where the light beam strikes the sample.

2.2.1.2. Clean sample cells by thorough washing with laboratory soap (inside and out) followed by multiple rinses with distilled or de-ionized water, and let air-dry.

2.2.2. Use a very thin layer of silicone oil on the outside surfaces to mask minor imperfections or scratches in the cells.

2.2.2.1. Use silicone oil with the same refractive index of the glass; making sure the cell appear to be nearly dry with little or no visible signs of oil.

2.2.3. Because small differences between cells significantly impact measurement, use either matched pairs or the same cell for standardization and sample measurement.

2.3. Standards:

2.3.1. Primary standards: Use these standards for initial calibration.

2.3.1.1. Formazin standards can be either obtained commercially or prepared according to method SM 2130B, section 3.b. See *Standard Methods for the Examination of Water and Wastewater* (American Public Health Association, American Water Works Association, Water Pollution Control Federation).

2.3.1.2. Some instruments may require the use of styrene divinylbenzene (SDVB) standards for calibration.

2.3.2. Secondary Standards: Use only those certified by the manufacturer for a specific instrument. Secondary standards must only be used for continuing calibration verifications according to the procedures in section 3.4 below. Determine or verify the values of secondary standards according to the procedure in section 3.3 below.

2.3.3. Turbidity-free water: Use filtered, laboratory reagent water demonstrated to be free of measurable turbidity (<0.01 NTU) or purchase commercially prepared turbidity-free water.

3. CALIBRATION AND USE

3.1. General Concerns

3.1.1. Light absorption by dissolved and suspended matter may cause a negative bias on the turbidity measurement. When present in significant concentrations, particles of light-absorbing materials such as activated carbon will cause a negative interference. Likewise, the presence of dissolved, color-causing substances that absorb light may also cause a negative interference. Some commercial instruments may have the capability of either correcting for slight color interference or optically blanking out the color effect.

3.1.2. Handle samples with natural effervescence as described in 3.5.5.1 below.

3.2. Calibration and Initial Calibration Verification

3.2.1. Follow the calibration activities in FT 1000, section 2.2.

3.2.2. Perform an initial calibration using at least two primary standards.

3.2.2.1. If the instrument cannot be calibrated with two standards, calibrate the instrument with one standard and verify with a second standard per 3.2.3 below.

3.2.2.2. For measurement of samples of very low turbidity, select the lowest standard commercially available for bracketing the lower end of the anticipated sample turbidity range or dilute higher turbidity standards with turbidity-free water.

3.2.2.3. Do not use turbidity-free water as a calibration verification standard.

3.2.3. Perform an initial calibration verification by reading at least one primary standard as a sample. The acceptance criterion for the initial calibration verification depends on the range of turbidity of the standard value:

- Standard Value = 0.1-10 NTU: the response must be within 10% of the standard;
- Standard Value = 11-40 NTU: the response must be within 8% of the standard;
- Standard Value = 41-100 NTU: the response must be within 6.5% of the standard; and
- Standard Value > 100 NTU: the response must be within 5% of the standard.

3.3. Determining the Values of Secondary Standards

3.3.1. Use only those standards certified by the manufacturer for a specific instrument.

3.3.2. Use verified secondary standards only for continuing calibration verifications.

3.3.3. Determining the initial value(s) of secondary standard(s):

3.3.3.1. Calibrate or verify the instrument with primary standards. Select primary standards that bracket the range of the secondary standards.

3.3.3.2. Immediately after the an initial calibration with primary standards or verification with a primary standard, read each secondary standard as a sample use the reading from the instrument as the first assigned value.

3.3.4. Verifying Secondary Standards

3.3.4.1. At least once per quarter or at other documented intervals (see 3.3.5 below), determine or verify the values of secondary standards immediately after the instrument has been calibrated or verified with primary standards.

3.3.4.2. Read each secondary standard as a sample. This reading must be within the manufacturer's stated tolerance range and within the acceptance ranges of the assigned standard value as listed in 3.2.3., above. If the criteria in section 3.2.3., above are not met, assign this reading as the value of the standard. If the reading is outside the manufacturer's stated tolerance range, discard the secondary standard.

3.3.5. **More frequent calibration verifications may be required for discharge permit compliance measurements or other regulatory requirements.**

3.4. Continuing Calibration Verification: Perform a continuing calibration verification using at least one primary or secondary standard. The calibration acceptance criteria are the same as those listed in section 3.2.3 above.

3.5. Measuring Turbidity in Samples

3.5.1. Gently agitate the sample and wait until air bubbles disappear.

3.5.2. Double-rinse the sample cell or cuvette with a small amount of the sample. Discard, and pour an aliquot into the sample cell or cuvette.

3.5.3. Gently dry out its external surface with lint-free paper.

3.5.4. Insert the cell in the instrument and read the turbidity directly from the meter display.

3.5.5. Do not use vacuum degassing, ultrasonic bath or other devices to remove bubbles from the sample. If the sample contains visible bubbles or if it effervesces (as in groundwater, with changes in pressure and temperature), make a note of this in the field records and collect a sample for laboratory measurement.

3.5.5.1. If effervescing samples are collected for laboratory analysis collect the sample without leaving headspace in the container and ship it as soon as possible to the laboratory (the holding time for this measurement is only 48 hrs). Ship this sample in wet ice at 4^{o}C.

3.5.6. Pour out the sample, double-rinse the cuvette with de-ionized water in preparation for the next sample.

4. PREVENTIVE MAINTENANCE: Refer to FT 1000, section 3.

5. DOCUMENTATION

5.1. Standard and Reagent Documentation: Document information about standards and reagents used for calibrations, verifications, and sample measurements.

5.1.1. Note the date of receipt, the expiration date and the date of first use for all standards and reagents.

5.1.1.1. Document acceptable verification of any standard used after its expiration date.

5.1.2. Record the concentration or other value for the standard in the appropriate measurement units.

5.1.2.1. Note vendor catalog number and description for preformulated solutions as well as for neat liquids and powdered standards.

5.1.2.2. Retain vendor assay specifications for standards as part of the calibration record.

5.1.3. Record the grade of standard or reagent used.

5.1.4. When formulated in-house, document all calculations used to formulate calibration standards.

5.1.4.1. Record the date of preparation for all in-house formulations.

5.1.5. Describe or cite the procedure(s) used to prepare any standards in-house (DEP SOP or internal SOP).

5.2. Field Instrument Calibration Documentation: Document acceptable calibration and calibration verification for each instrument unit and field test or analysis, linking this record with affected sample measurements.

5.2.1. Retain vendor certifications of all factory-calibrated instrumentation.

5.2.2. Designate the identity of specific instrumentation in the documentation with a unique description or code for each instrument unit used.

5.2.2.1. Record manufacturer name, model number, and identifying number (such as a serial number) for each instrument unit.

5.2.3. Record the time and date of all initial calibrations and all calibration verifications.

5.2.4. Record the instrument reading (value in appropriate measurement units) of all calibration verifications.

5.2.5. Record the name of the analyst(s) performing the calibration.

5.2.6. Document the specific standards used to calibrate or verify the instrument or field test with the following information:

- Type of standard or standard name (e.g., formazin)
- Value of standard, including correct units (e.g., 20 NTU)
- Link to information recorded according to section 5.1 above

5.2.7. Retain manufacturers' instrument specifications.

5.2.8. Document whether successful initial calibration occurred.

5.2.9. Document whether each calibration verification passed or failed.

5.2.10. Document any corrective actions taken to correct instrument performance according to records requirements of FD 3000.

5.2.10.1. Document date and time of any corrective action.

5.2.10.2. Note any incidence of discontinuation of use of the instrument due to calibration failure.

5.2.11. Describe or cite the specific calibration or verification procedure performed (DEP SOP or internal SOP).

5.3. Record all field-testing measurement data, to include the following:

- Project name
- Date and time of measurement or test (including time zone, if applicable)
- Source and location of the measurement or test sample (e.g., monitoring well identification number, outfall number, station number or other description)
- Latitude and longitude of sampling source location (if required)
- Analyte or parameter measured
- Measurement or test sample value
- Reporting units
- Initials or name of analyst performing the measurement
- Unique identification of the specific instrument unit(s) used for the test(s)

APPENDIX D

LABORATORY STANDARD OPERATING PROCEDURES AND DoD ELAP ACCREDITATION

LABORATORY ACCREDITATION BUREAU

Certificate Number L2223

Certificate of Accreditation

Accredited to DoD ELAP and ISO/IEC 17025:2005

Katahdin Analytical Services, Inc.

600 Technology Way
Scarborough, ME 04074

has met the requirements set forth in L-A-B's policies and procedures, all requirements of ISO/IEC 17025:2005 "General Requirements for the competence of Testing and Calibration Laboratories" and the U.S. Department of Defense Environmental Laboratory Accreditation Program (DoD ELAP).*

The accredited lab has demonstrated technical competence to a defined "Scope of Accreditation" and the operation of a laboratory quality management system (refer to joint ISO-ILAC-IAF Communiqué dated 8 January 2009).

Accreditation Granted through: November 4, 2012

R. Douglas Leonard, Jr., Managing Director
Laboratory Accreditation Bureau
Presented the 4th of November, 2009

*See the laboratory's Scope of Accreditation for details of the DoD ELAP requirements
Laboratory Accreditation Bureau is found to be in compliance with ISO/IEC 17011:2004 and recognized by ILAC (International Laboratory Accreditation Cooperation) and NACLA (National Cooperation for Laboratory Accreditation).

LABORATORY ACCREDITATION BUREAU

Certificate # L2223

Scope of Accreditation For Katahdin Analytical Services

600 Technology Way
Scarborough, ME 04074
Leslie Dimond
1- 207-874-2400

In recognition of a successful assessment to ISO/IEC 17025:2005 and the requirements of the DoD Environmental Laboratory Accreditation Program (DoD ELAP) as detailed in the DoD Quality Systems Manual for Environmental Laboratories (DoD QSM v4.1) based on the National Environmental Laboratory Accreditation Conference Chapter 5 Quality Systems Standard (NELAC Voted Revision June 5, 2003), accreditation is granted to Katahdin Analytical Services to perform the following tests:

Accreditation granted through: **November 4, 2012**

Testing - Environmental

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/ECD	608 / 8081A,B/ SOM01.2	4 4`-DDD
GC/ECD	608 / 8081A,B/ SOM01.2	4 4`-DDE
GC/ECD	608 / 8081A,B / SOM01.2	4 4`-DDT
GC/ECD	608 / 8081A,B / SOM01.2	Aldrin
GC/ECD	608 / 8081A,B / SOM01.2	alpha-BHC (alpha-Hexachlorocyclohexane)
GC/ECD	8081A,B / SOM01.2	Alpha-Chlordane
GC/ECD	608 / 8081A,B / SOM01.2	beta-BHC (beta-Hexachlorocyclohexane)
GC/ECD	608 /8081A,B	Chlordane (tech.)
GC/ECD	608 / 8081A,B / SOM01.2	delta-BHC
GC/ECD	608 / 8081A,B / SOM01.2	Dieldrin
GC/ECD	608 / 8081A,B / SOM01.2	Endosulfan I
GC/ECD	608 / 8081A,B / SOM01.2	Endosulfan II
GC/ECD	608 / 8081A,B / SOM01.2	Endosulfan sulfate
GC/ECD	608 / 8081A,B / SOM01.2	Endrin
GC/ECD	608 / 8081A,B / SOM01.2	Endrin aldehyde
GC/ECD	8081A,B / SOM01.2	Endrin Ketone
GC/ECD	8081A,B / SOM01.2	gamma-BHC (Lindane gamma-Hexachlorocyclohexane)
GC/ECD	608 / 8081A,B / SOM01.2	Heptachlor
GC/ECD	608 / 8081A,B / SOM01.2	Heptachlor epoxide

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/ECD	8081A,B / SOM01.2	Methoxychlor
GC/ECD	608 / 8081A,B / SOM01.2	Toxaphene (Chlorinated camphene)
GC/ECD	608 / 8082/8082A / SOM01.2	Aroclor-1221 (PCB-1221)
GC/ECD	608 / 8082/8082A / SOM01.2	Aroclor-1232 (PCB-1232)
GC/ECD	608 / 8082/8082A / SOM01.2	Aroclor-1242 (PCB-1242)
GC/ECD	608 / 8082/8082A / SOM01.2	Aroclor-1248 (PCB-1248)
GC/ECD	608 / 8082/8082A / SOM01.2	Aroclor-1254 (PCB-1254)
GC/ECD	608 / 8082/8082A / SOM01.2	Aroclor-1260 (PCB-1260)
GC/ECD	8082/8082A	Aroclor-1262 (PCB-1262)
GC/ECD	8082/8082A	Aroclor-1268 (PCB-1268)
GC/ECD	8082/8082A	2 2' 3 3' 4 4' 5 5' 6-Nonachlorobiphenyl (BZ 206)
GC/ECD	8082/8082A	2 2' 3 3' 4 4' 5 6-Octachlorobiphenyl (BZ 195)
GC/ECD	8082/8082A	2 2' 3 3' 4 4' 5-Heptachlorobiphenyl (BZ 170)
GC/ECD	8082/8082A	2 2' 3 3' 4 4'-Hexachlorobiphenyl (BZ 128)
GC/ECD	8082/8082A	2 2' 3 4 4' 5 5'-Heptachlorobiphenyl (BZ 180)
GC/ECD	8082/8082A	2 2' 3 4 4' 5' 6-Heptachlorobiphenyl (BZ 183)
GC/ECD	8082/8082A	2 2' 3 4 4' 5'-Hexachlorobiphenyl (BZ 138)
GC/ECD	8082/8082A	2 2' 3 4 4' 6 6'-Heptachlorobiphenyl (BZ 184)
GC/ECD	8082/8082A	2 2' 3 4' 5 5' 6-Heptachlorobiphenyl (BZ 187)
GC/ECD	8082/8082A	2 2' 3 4 5'-Pentachlorobiphenyl (BZ 87)
GC/ECD	8082/8082A	2 2' 3 5'-Tetrachlorobiphenyl (BZ 44)
GC/ECD	8082/8082A	2 2' 4 4' 5 5'-Hexachlorobiphenyl (BZ 153)
GC/ECD	8082/8082A	2 2' 4 5 5'-Pentachlorobiphenyl (BZ 101)
GC/ECD	8082/8082A	2 2' 4' 5-Tetrachlorobiphenyl (BZ 49)
GC/ECD	8082/8082A	2 2' 5 5'-Tetrachlorobiphenyl (BZ 52)
GC/ECD	8082/8082A	2 2' 5-Trichlorobiphenyl (BZ 18)
GC/ECD	8082/8082A	2 3 3' 4 4' 5-Hexachlorobiphenyl (BZ 156)
GC/ECD	8082/8082A	2 3 3' 4 4' 5'-Hexachlorobiphenyl (BZ 157)
GC/ECD	8082/8082A	2 3 3' 4 4'-Pentachlorobiphenyl (BZ 105)
GC/ECD	8082/8082A	2 3 3' 4 4' 5 5'-Heptachlorobiphenyl (BZ 189)
GC/ECD	8082/8082A	2 3' 4 4' 5 5'-Hexachlorobiphenyl (BZ 167)
GC/ECD	8082/8082A	2 3' 4 4' 5-Pentachlorobiphenyl (BZ 118)
GC/ECD	8082/8082A	2 3' 4 4'5-Pentachlorobiphenyl (BZ 123)
GC/ECD	8082/8082A	2 3' 4 4'-Tetrachlorobiphenyl (BZ 66)
GC/ECD	8082/8082A	2 3' 4 4' 5-Pentachlorobiphenyl (BZ 114)
GC/ECD	8082/8082A	2 4 4'-Trichlorobiphenyl (BZ 28)
GC/ECD	8082/8082A	2 4'-Dichlorobiphenyl (BZ 8)
GC/ECD	8082/8082A	3 3' 4 4' 5 5'-Hexachlorobiphenyl (BZ 169)
GC/ECD	8082/8082A	3 3' 4 4' 5-Pentachlorobiphenyl (BZ 126)
GC/ECD	8082/8082A	3 3' 4 4'-Tetrachlorobiphenyl (BZ 77)
GC/ECD	8082/8082A	3 4 4' 5-Tetrachlorobiphenyl (BZ 81)
GC/ECD	8082/8082A	Decachlorobiphenyl (BZ 209)
GC/ECD	8151A	2 4 5-T
GC/ECD	8151A	2 4-D
GC/ECD	8151A	2 4-DB

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/ECD	8151A	Dalapon
GC/ECD	8151A	Dicamba
GC/ECD	8151A	Dichloroprop
GC/ECD	8151A	DInoseb
GC/ECD	8151A	MCPA
GC/ECD	8151A	MCPP
GC/ECD	8151A	Pentachlorophenol
GC/ECD	8151A	Silvex (2 4 5-TP)
GC/FID	8015B/C	Diesel range organics (DRO)
GC/FID	8015B/C	Gasoline range organics (GRO)
GC/FID	8011 / 504	1 2-Dibromoethane (EDB)
GC/FID	8011 / 504	1 2-Dibromo-3-chloropropane
GC/FID	RSK-175	Methane Ethane Ethene
GC/MS	8260B,C / 524.2	1 1 1 2-Tetrachloroethane
GC/MS	624 / 8260B,C / SOM01.2 / 524.2	1 1 1-Trichloroethane
GC/MS	624 / 8260B,C / SOM01.2 / 524.2	1 1 2 2-Tetrachloroethane
GC/MS	SOM01.2	1 1 2-Trichloro-1 2 2-trifluoroethane
GC/MS	624 / 8260B,C / SOM01.2 / 524.2	1 1 2-Trichloroethane
GC/MS	624 / 8260B,C / SOM01.2 / 524.2	1 1-Dichloroethane
GC/MS	624 / 8260B,C / SOM01.2 / 524.2	1 1-Dichloroethene
GC/MS	8260B,C / 524.2	1 1-Dichloropropene
GC/MS	8260B,C / SOM01.2 / 524.2	1 2 3-Trichlorobenzene
GC/MS	8260B,C / 524.2	1 2 3-Trichloropropane
GC/MS	8260B,C / SOM01.2 / 524.2	1 2 4-Trichlorobenzene
GC/MS	8260B,C / 524.2	1 2 4-Trimethylbenzene
GC/MS	8260B,C / SOM01.2 / 524.2	1 2-Dibromo-3-chloropropane
GC/MS	8260B,C / SOM01.2 / 524.2	1 2-Dibromoethane (EDB)
GC/MS	624 / 8260B,C / SOM01.2 / 524.2	1 2-Dichlorobenzene
GC/MS	624 / 8260B,C / SOM01.2 / 524.2	1 2-Dichloroethane
GC/MS	624 / 8260B,C / SOM01.2 / 524.2	1 2-Dichloropropane
GC/MS	8260B,C / 524.2	1 3 5-Trimethylbenzene
GC/MS	624 / 8260B,C / SOM01.2 / 524.2	1 3-Dichlorobenzene
GC/MS	8260B,C / 524.2	1 3-Dichloropropane
GC/MS	624 / 8260B,C / SOM01.2 / 524.2	1 4-Dichlorobenzene
GC/MS	8260B,C / SOM01.2	1 4-Dioxane
GC/MS	8260B,C / 524.2	2 2-Dichloropropane

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS	8260B,C / SOM01.2 / 524.2	2-Butanone
GC/MS	624 / 8260B,C	2-Chloroethyl vinyl ether
GC/MS	8260B,C / 524.2	2-Chlorotoluene
GC/MS	8260B,C / SOM01.2 / 524.2	2-Hexanone
GC/MS	8260B,C / 524.2	4-Chlorotoluene
GC/MS	8260B,C / SOM01.2 / 524.2	4-Methyl-2-pentanone
GC/MS	8260B,C / SOM01.2 / 524.2	Acetone
GC/MS	8260B,C	Acetonitrile
GC/MS	624 / 8260B,C	Acrolein
GC/MS	624 / 8260B,C / 524.2	Acrylonitrile
GC/MS	8260B,C / 524.2	Allyl chloride
GC/MS	624 / 8260B,C / SOM01.2 / 524.2	Benzene
GC/MS	8260B,C / 524.2	Bromobenzene
GC/MS	8260B,C / SOM01.2 / 524.2	Bromochloromethane
GC/MS	624 / 8260B,C / SOM01.2 / 524.2	Bromodichloromethane
GC/MS	624 / 8260B,C / SOM01.2 / 524.2	Bromoform
GC/MS	8260B,C / SOM01.2 / 524.2	Carbon disulfide
GC/MS	624 / 8260B,C / SOM01.2 / 524.2	Carbon tetrachloride
GC/MS	624 / 8260B,C / SOM01.2 / 524.2	Chlorobenzene
GC/MS	624 / 8260B,C / SOM01.2 / 524.2	Chloroethane
GC/MS	624 / 8260B,C / SOM01.2 / 524.2	Chloroform
GC/MS	8260B,C	Chloroprene
GC/MS	8260B,C / SOM01.2 / 524.2	cis-1 2-Dichloroethene
GC/MS	624 / 8260B,C / SOM01.2 / 524.2	cis-1 3-Dichloropropene
GC/MS	SOM01.2	Cyclohexane
GC/MS	624 / 8260B,C / SOM01.2 / 524.2	Dibromochloromethane
GC/MS	8260B,C / 524.2	Dibromomethane
GC/MS	624 / 8260B,C / SOM01.2 / 524.2	Dichlorodifluoromethane
GC/MS	8260B,C / 524.2	Diethyl ether
GC/MS	8260B,C	Di-isopropylether
GC/MS	8260B,C / 524.2	Ethyl methacrylate
GC/MS	624 / 8260B,C / SOM01.2 / 524.2	Ethylbenzene
GC/MS	8260B,C	Ethyl-t-butylether
GC/MS	8260B,C / 524.2	Hexachlorobutadiene
GC/MS	8260B,C	Iodomethane
GC/MS	8260B,C	Isobutyl alcohol

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS	8260B,C / SOM01.2 / 524.2	Isopropyl benzene
GC/MS	8260B,C / SOM01.2 / 524.2	m p-xylenes
GC/MS	8260B,C / 524.2	Methacrylonitrile
GC/MS	SOM01.2	Methyl acetate
GC/MS	624 / 8260B,C / SOM01.2 / 524.2	Methyl bromide (Bromomethane)
GC/MS	624 / 8260B,C / SOM01.2 / 524.2	Methyl chloride (Chloromethane)
GC/MS	8260B,C / 524.2	Methyl methacrylate
GC/MS	8260B,C / SOM01.2 / 524.2	Methyl tert-butyl ether
GC/MS	SOM01.2	Methylcyclohexane
GC/MS	624 / 8260B,C / SOM01.2 / 524.2	Methylene chloride
GC/MS	8260B,C / 524.2	Naphthalene
GC/MS	8260B,C / 524.2	n-Butylbenzene
Gc/ms	8260B,C / 524.2	n-Propylbenzene
GC/MS	8260B,C / SOM01.2 / 524.2	o-Xylene
GC/MS	8260B,C / 524.2	p-Isopropyltoluene
GC/MS	8260B,C / 524.2	Propionitrile
GC/MS	8260B,C / 524.2	sec-butylbenzene
GC/MS	8260B,C / SOM01.2 / 524.2	Styrene
GC/MS	8260B,C	t-Amylmethylether
GC/MS	8260B,C / 524.2	tert-Butyl alcohol
GC/MS	8260B,C	tert-Butylbenzene
GC/MS	624 / 8260B,C / SOM01.2 / 524.2	Tetrachloroethene (Perchloroethylene)
GC/MS	8260B,C / 524.2	Tetrahydrofuran
GC/MS	624 / 8260B,C / SOM01.2 / 524.2	Toluene
GC/MS	624 / 8260B,C / SOM01.2 / 524.2	trans-1 2-Dichloroethylene
GC/MS	624 / 8260B,C / SOM01.2 / 524.2	trans-1 3-Dichloropropylene
GC/MS	8260B,C / 524.2	trans-1 4-Dichloro-2-butuene
GC/MS	624 / 8260B,C / SOM01.2 / 524.2	Trichloroethene (Trichloroethylene)
GC/MS	624 / 8260B,C / SOM01.2 / 524.2	Trichlorofluoromethane
GC/MS	8260B,C	Vinyl acetate
GC/MS	624 / 8260B,C / SOM01.2 / 524.2	Vinyl chloride
GC/MS	624 8260B,C	Xylene
GC/MS	8270C,D / SOM01.2	1 2 4 5-Tetrachlorobenzene
GC/MS	625 / 8270C,D / SOM01.2	1 2 4-Trichlorobenzene
GC/MS	625 / 8270C,D / SOM01.2	1 2-Dichlorobenzene
GC/MS	8270C,D	1 2-Diphenylhydrazine

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS	8270C,D	1 3 5-Trinitrobenzene
GC/MS	625 / 8270C,D / SOM01.2	1 3-Dichlorobenzene
GC/MS	8270C,D	1 3-Dinitrobenzene
GC/MS	625 / 8270C,D / SOM01.2	1 4-Dichlorobenzene
GC/MS	8270C,D	1 4-Dioxane
GC/MS	8270C,D	1 4-Naphthoquinone
GC/MS	8270C,D	1 4-Phenylenediamine
GC/MS	8270C,D	1-Naphthylamine
GC/MS	8270C,D / SOM01.2	2 3 4 6-Tetrachlorophenol
GC/MS	8270C,D / SOM01.2	2 4 5-Trochlorophenol
GC/MS	625 / 8270C,D / SOM01.2	2 4 6-Trichlorophenol
GC/MS	625 / 8270C,D / SOM01.2	2 4-Dichlorophenol
GC/MS	625 / 8270C,D / SOM01.2	2 4-Dimethylphenol
GC/MS	625 / 8270C,D / SOM01.2	2 4-Dinitrophenol
GC/MS	625 / 8270C,D / SOM01.2	2 4-Dinitrotoluene (2 4-DNT)
GC/MS	8270C,D	2 6-Dichlorophenol
GC/MS	625 / 8270C,D / SOM01.2	2 6-Dinitrotoluene (2 6-DNT)
GC/MS	8270C,D	2-Acetylaminofluorene
GC/MS	625 / 8270C,D / SOM01.2	2-Chloronaphthalene
GC/MS	625 / 8270C,D / SOM01.2	2-Chlorophenol
GC/MS	625 / 8270C,D / SOM01.2	2-Methyl-4 6-dinitrophenol
GC/MS	8270C,D / SOM01.2	2-Methylnaphthalene
GC/MS	8270C,D / SOM01.2	2-Methylphenol
GC/MS	8270C,D	2-Naphthylamine
GC/MS	8270C,D	2-Nitroaniline
GC/MS	625 / 8270C,D / SOM01.2	2-Nitrophenol
GC/MS	8270C,D	2-Picoline
GC/MS	625 / 8270C,D / SOM01.2	3 3'-Dichlorobenzidine
GC/MS	8270C,D	3 3'-Dimethylbenzidine
GC/MS	8270C,D	3-Methylcholanthrene
GC/MS	8270C,D / SOM01.2	3-Nitroaniline
GC/MS	8270C,D	4-Aminobiphenyl
GC/MS	625 / 8270C,D / SOM01.2	4-Bromophenyl phenyl ether
GC/MS	625 / 8270C,D / SOM01.2	4-Chloro-3-methylphenol
GC/MS	8270C,D / SOM01.2	4-Chloroaniline
GC/MS	625 / 8270C,D / SOM01.2	4-Chlorophenyl phenylether
GC/MS	8270C,D	4-Dimethyl aminoazobenzene
GC/MS	8270C,D / SOM01.2	4-Methylphenol
GC/MS	8270C,D / SOM01.2	4-Nitroaniline
GC/MS	625 / 8270C,D / SOM01.2	4-Nitrophenol
GC/MS	8270C,D	5-Nitro-o-toluidine
GC/MS	8270C,D	7,12-Dimethylphenethylamine
GC/MS	8270C,D	a a-Dimethylphenethylamine
GC/MS	625 / 8270C,D / SOM01.2	Acenaphthene
GC/MS	625 / 8270C,D / SOM01.2	Acenaphthylene

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS	8270C,D / SOM01.2	Acetophenone
GC/MS	8270C,D	Aniline
GC/MS	625 / 8270C,D / SOM01.2	Anthracene
GC/MS	8270C,D	Aramite
GC/MS	8270C,D / SOM01.2	Atrazine
GC/MS	SOM01.2	Benzaldehyde
GC/MS	625 / 8270C,D	Benzidine
GC/MS	625 / 8270C,D / SOM01.2	Benzo(a)anthracene
GC/MS	625 / 8270C,D / SOM01.2	Benzo(a)pyrene
GC/MS	625 / 8270C,D / SOM01.2	Benzo(b)fluoranthene
GC/MS	625 / 8270C,D / SOM01.2	Benzo(g h i)perylene
GC/MS	625 / 8270C,D / SOM01.2	Benzo(k)fluoranthene
GC/MS	8270C,D	Benzoic Acid
GC/MS	8270C,D	Benzyl alcohol
GC/MS	8270C,D / SOM01.2	Biphenyl
GC/MS	625 / 8270C,D / SOM01.2	bis(2-Chloroethoxy)methane
GC/MS	625 / 8270C,D / SOM01.2	bis(2-Chloroethyl) ether
GC/MS	625 / 8270C,D / SOM01.2	bis(2-Chloroisopropyl) ether (2 2`-Oxybis(1-chloropropane))
GC/MS	625 / 8270C,D / SOM01.2	bis(2-Ethylhexyl) phthalate (DEHP)
GC/MS	625 / 8270C,D / SOM01.2	Butyl benzyl phthalate
GC/MS	SOM01.2	Caprolactam
GC/MS	8270C,D / SOM01.2	Carbazole
GC/MS	8270C,D	Chlorobenzilate
GC/MS	625 / 8270C,D / SOM01.2	Chrysene
GC/MS	8270C,D	Diallate
GC/MS	625 / 8270C,D / SOM01.2	Dibenz(a h)anthracene
GC/MS	8270C,D / SOM01.2	Dibenzofuran
GC/MS	625 / 8270C,D / SOM01.2	Diethyl phthalate
GC/MS	8270C,D	Dimethoate
GC/MS	625 / 8270C,D / SOM01.2	Dimethyl phthalate
GC/MS	625 / 8270C,D / SOM01.2	Di-n-butyl phthalate
GC/MS	625 / 8270C,D / SOM01.2	Di-n-octyl phthalate
GC/MS	8270C,D	Ethyl methanesulfonate
GC/MS	8270C,D	Famfur
GC/MS	625 / 8270C,D / SOM01.2	Fluoranthene
GC/MS	625 / 8270C,D / SOM01.2	Fluorene
GC/MS	625 / 8270C,D / SOM01.2	Hexachlorobenzene
GC/MS	625 / 8270C,D / SOM01.2	Hexachlorobutadiene
GC/MS	625 / 8270C,D / SOM01.2	Hexachlorocyclopentadiene
GC/MS	625 / 8270C,D / SOM01.2	Hexachloroethane
GC/MS	8270C,D	Hexachloropropene
GC/MS	625 / 8270C,D / SOM01.2	Indeno(1 2 3-cd)pyrene
GC/MS	8270C,D	Isodrin
GC/MS	625 / 8270C,D / SOM01.2	Isophorone

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS	8270C,D	Isosafrole
GC/MS	8270C,D	Methapyriline
GC/MS	8270C,D	Methy methanesulfonate
GC/MS	8270C,D	Methyl parathion
GC/MS	625 / 8270C,D / SOM01.2	Naphthalene
GC/MS	625 / 8270C,D / SOM01.2	Nitrobenzene
GC/MS	8270C,D	Nitroquinoline-1-oxide
GC/MS	8270C,D	n-Nitrosodiethylamine
GC/MS	625 / 8270C,D / SOM01.2	n-Nitrosodimethylamine
GC/MS	8270C,D	n-Nitroso-di-n-butylamine
GC/MS	625 / 8270C,D / SOM01.2	n-Nitrosodi-n-propylamine
GC/MS	625 / 8270C,D / SOM01.2	n-Nitrosodiphenylamine
GC/MS	8270C,D	n-Nitrosomethylethylamine
GC/MS	8270C,D	n-Nitrosomorpholine
GC/MS	8270C,D	n-Nitrosopiperidine
GC/MS	8270C,D	n-Nitrosopyrrolidine
GC/MS	8270C,D	o o o-Triethyl phosphorothioate
GC/MS	8270C,D	o-Toluidine
GC/MS	8270C,D	Pentachlorobenzene
GC/MS	8270C,D	Pentachloronitrobenzene
GC/MS	625 / 8270C,D / SOM01.2	Pentachlorophenol
GC/MS	8270C,D	Phenacetin
GC/MS	625 / 8270C,D / SOM01.2	Phenanthrene
GC/MS	625 / 8270C,D / SOM01.2	Phenol
GC/MS	8270C,D	Phorate
GC/MS	8270C,D	Pronamide
GC/MS	625 / 8270C,D / SOM01.2	Pyrene
GC/MS	8270C,D	Pyrididne
GC/MS	8270C,D	Safrole
GC/MS	8270C,D	Thionazin
HPLC	8330/8330A/8330B	1 3 5-Trinitrobenzene
HPLC	8330/8330A/8330B	1 3-Dinitrobenzene
HPLC	8330/8330A/8330B	2 4 6-Trinitrotoluene
HPLC	8330/8330A/8330B	2 4-Dinitrotoluene
HPLC	8330/8330A/8330B	2 6-Dinitrotoluene
HPLC	8330/8330A/8330B	2-Amino-4 6 -dinitrotoluene
HPLC	8330/8330A/8330B	2-Nitrotoluene
HPLC	8330/8330A/8330B	3-Nitrotoluene
HPLC	8330/8330A/8330B	4-Amino-2,3-dinitrotoluene
HPLC	8330/8330A/8330B	4-Nitrotoluene
HPLC	8330/8330A/8330B	Hexahydro-1 3 5-trinitro-1 3 5-triazine (RDX)
HPLC	8330/8330A/8330B	Nitrobenzene
HPLC	8330/8330A/8330B	Nitroglycerin
HPLC	8330/8330A/8330B	Octahydro-1 3 5 7-tetrazocine (HMX)
HPLC	8330/8330A/8330B	Tetryl

Non-Potable Water		
Technology	**Method**	**Analyte**
CVAA	245.1 / 7470A / ILM05.3	Mercury
CVAF	1631E	Low Level Mercury
ICP	200.7 / 6010B,C / ILM05.3	Aluminum
ICP	200.7 / 6010B,C / ILM05.3	Antimony
ICP	200.7 / 6010B,C / ILM05.3	Arsenic
ICP	200.7 / 6010B,C / ILM05.3	Barium
ICP	200.7 / 6010B,C / ILM05.3	Beryllium
ICP	200.7 / 6010B,C	Boron
ICP	200.7 / 6010B,C / ILM05.3	Cadmium
ICP	200.7 / 6010B,C / ILM05.3	Calcium
ICP	200.7 / 6010B,C / ILM05.3	Chromium
ICP	200.7 / 6010B,C / ILM05.3	Cobalt
ICP	200.7 / 6010B,C / ILM05.3	Copper
ICP	200.7 / 6010B,C / ILM05.3	Iron
ICP	200.7 / 6010B,C / ILM05.3	Lead
ICP	200.7 / 6010B,C / ILM05.3	Magnesium
ICP	200.7 / 6010B,C / ILM05.3	Manganese
ICP	200.7 / 6010B,C	Molybdenum
ICP	200.7 / 6010B,C / ILM05.3	Nickel
ICP	200.7 / 6010B,C / ILM05.3	Potassium
ICP	200.7 / 6010B,C / ILM05.3	Selenium
ICP	200.7	Silicon
ICP	200.7 / 6010B,C / ILM05.3	Silver
ICP	200.7 / 6010B,C / ILM05.3	Sodium
ICP	6010B,C	Strontium
ICP	200.7 / 6010B,C / ILM05.3	Thallium
ICP	200.7 / 6010B,C	Tin
ICP	200.7 / 6010B,C	Titanium
ICP	200.7 / 6010B,C / ILM05.3	Vanadium
ICP	200.7 / 6010B,C / ILM05.3	Zinc
ICP/MS	200.8 / 6020/6020A / ILM05.3	Aluminum
ICP/MS	200.8 / 6020/6020A / ILM05.3	Antimony
ICP/MS	200.8 / 6020/6020A / ILM05.3	Arsenic
ICP/MS	200.8 / 6020/6020A / ILM05.3	Barium
ICP/MS	200.8 / 6020/6020A / ILM05.3	Beryllium
ICP/MS	200.8 / 6020/6020A	Boron
ICP/MS	200.8 / 6020/6020A / ILM05.3	Cadmium
ICP/MS	200.8 / 6020/6020A	Calcium
ICP/MS	200.8 / 6020/6020A / ILM05.3	Chromium
ICP/MS	200.8 / 6020/6020A / ILM05.3	Cobalt
ICP/MS	200.8 / 6020/6020A / ILM05.3	Copper
ICP/MS	200.8 / 6020/6020A	Iron
ICP/MS	200.8 / 6020/6020A / ILM05.3	Lead
ICP/MS	200.8 / 6020/6020A / ILM05.3	Magnesium
ICP/MS	200.8 / 6020/6020A	Manganese

Non-Potable Water		
Technology	**Method**	**Analyte**
ICP/MS	200.8 / 6020/6020A	Molybdenum
ICP/MS	200.8 / 6020/6020A / ILM05.3	Nickel
ICP/MS	200.8 / 6020/6020A	Potassium
ICP/MS	200.8 / 6020/6020A / ILM05.3	Selenium
ICP/MS	200.8 / 6020/6020A / ILM05.3	Silicon
ICP/MS	200.8 / 6020/6020A / ILM05.3	Silver
ICP/MS	200.8 / 6020/6020A	Sodium
ICP/MS	6020/6020A	Strontium
ICP/MS	200.8 / 6020/6020A / ILM05.3	Thallium
ICP/MS	200.8 / 6020/6020A	Tin
ICP/MS	200.8 / 6020/6020A	Titanium
ICP/MS	200.8	Uranium
ICP/MS	200.8 / 6020/6020A / ILM05.3	Vanadium
ICP/MS	200.8 / 6020/6020A / ILM05.3	Zinc
IC	300.0 / 9056/9056A	Bromide
IC	300.0 / 9056/9056A	Chloride
IC	300.0 / 9056/9056A	Nitrate as N
IC	300.0 / 9056/9056A	Nitrite as N
IC	300.0 / 9056/9056A	Nitrate + Nitrite
IC	300.0 / 9056/9056A	Orthophosphate as P
IC	300.0 / 9056/9056A	Sulfate
Titration	310.2 / 2320B	Alkalinity
Caculation	2340C	Hardness
Gravimetric	1664A	Oil and Grease
Gravimetric	2540 B, C, D	Solids
ISE	120.1 / 2510 B	Conductivity
ISE	2520B	Practical Salinity
ISE	4500F- C	Fluoride
ISE	4500H+ B	pH
ISE	5210B	TBOD / CBOD
Physical	1010 A	Ignitability
Physical	9040C	pH
Titration	2340B	Hardness
Titration	4500SO_3 B	Sulfite
Titration	9034 / 4500S^{2-} E	Sulfide
Titration	Chap. 7.3.4	Reactive Sulfide
TOC	9060A / 5310B	Total organic carbon
Turbidimetric	180.1 / 2130B	Turbidity
Turbidimetric	9038 / ASTM 516-02	Sulfate
UV/VIS	335.4 / 9012B / 4500-CN G	Amenable cyanide
UV/VIS	350.1 / 4500NH3 H	Ammonia as N
UV/VIS	3500Fe D	Ferrous Iron
UV/VIS	351.2	Kjeldahl nitrogen - total
UV/VIS	353.2 / 4500NO3 F	Nitrate + Nitrite

Non-Potable Water		
Technology	**Method**	**Analyte**
UV/VIS	353.2 / 4500NO3 F	Nitrate as N
UV/VIS	353.2 / 4500NO3 F	Nitrite as N
UV/VIS	365.1 / 4500P E	Orthophosphate as P
UV/VIS	365.4	Phosphorus total
UV/VIS	376.3	AVS-SEM
UV/VIS	410.4	COD
UV/VIS	420.1 / 9065	Total Phenolics
UV/VIS	4500Cl G	Total Residual Chlorine
UV/VIS	5540C	MBAS
UV/VIS	7196A / 3500-Cr D	Chromium VI
UV/VIS	9012B / ILM05.3/ 335.4	Total Cyanide
UV/VIS	9251 / 4500Cl E	Chloride
UV/VIS	Chap. 7.3.4	Reactive Cyanide
Preparation	**Method**	**Type**
Cleanup Methods	3640A	Gel Permeation Clean-up
Cleanup Methods	3630C	Silica Gel
Cleanup Methods	3660B	Sulfur Clean-Up
Cleanup Methods	3665A	Sulfuric Acid Clean-Up
Organic Preparation	3510C	Separatory Funnel Extraction
Organic Preparation	3520C	Continuous Liquid-Liquid Extraction
Inorganic Preparation	3010A	Hotblock
Volatile Organic Preparation	5030B,C	Purge and Trap

Solid and Chemical Waste		
Technology	**Method**	**Analyte**
GC/ECD	8081A,B/ SOM01.2	4 4`-DDD
GC/ECD	8081A,B / SOM01.2	4 4`-DDE
GC/ECD	8081A,B / SOM01.2	4 4`-DDT
GC/ECD	8081A,B / SOM01.2	Aldrin
GC/ECD	8081A,B / SOM01.2	alpha-BHC (alpha-Hexachlorocyclohexane)
GC/ECD	8081A,B / SOM01.2	Alpha-Chlordane
GC/ECD	8081A,B / SOM01.2	beta-BHC (beta-Hexachlorocyclohexane)
GC/ECD	608 /8081A,B	Chlordane (tech.)
GC/ECD	8081A,B / SOM01.2	delta-BHC
GC/ECD	8081A,B / SOM01.2	Dieldrin
GC/ECD	8081A,B / SOM01.2	Endosulfan I
GC/ECD	8081A,B / SOM01.2	Endosulfan II
GC/ECD	8081A,B / SOM01.2	Endosulfan sulfate
GC/ECD	8081A,B / SOM01.2	Endrin
GC/ECD	8081A,B / SOM01.2	Endrin aldehyde
GC/ECD	8081A,B / SOM01.2	Endrin Ketone

Solid and Chemical Waste		
Technology	**Method**	**Analyte**
GC/ECD	8081A,B / SOM01.2	gamma-BHC (Lindane gamma-Hexachlorocyclohexane)
GC/ECD	8081A,B / SOM01.2	Heptachlor
GC/ECD	8081A,B / SOM01.2	Heptachlor epoxide
GC/ECD	8081A,B / SOM01.2	Methoxychlor
GC/ECD	8081A,B / SOM01.2	Toxaphene (Chlorinated camphene)
GC/ECD	8082/8082A/ SOM01.2	Aroclor-1016 (PCB-1016)
GC/ECD	8082/8082A/ SOM01.2	Aroclor-1221 (PCB-1221)
GC/ECD	8082/8082A/ SOM01.2	Aroclor-1232 (PCB-1232)
GC/ECD	8082/8082A/ SOM01.2	Aroclor-1242 (PCB-1242)
GC/ECD	8082/8082A/ SOM01.2	Aroclor-1248 (PCB-1248)
GC/ECD	8082/8082A/ SOM01.2	Aroclor-1254 (PCB-1254)
GC/ECD	8082/8082A/ SOM01.2	Aroclor-1260 (PCB-1260)
GC/ECD	8082/8082A	Aroclor-1262 (PCB-1262)
GC/ECD	8082/8082A	Aroclor-1268 (PCB-1268)
GC/ECD	8082/8082A	2 2' 3 3' 4 4' 5 5' 6-Nonachlorobiphenyl (BZ 206)
GC/ECD	8082/8082A	2 2' 3 3' 4 4' 5 6-Octachlorobiphenyl (BZ 195)
GC/ECD	8082/8082A	2 2' 3 3' 4 4' 5-Heptachlorobiphenyl (BZ 170)
GC/ECD	8082/8082A	2 2' 3 3' 4 4'-Hexachlorobiphenyl (BZ 128)
GC/ECD	8082/8082A	2 2' 3 4 4' 5 5'-Heptachlorobiphenyl (BZ 180)
GC/ECD	8082/8082A	2 2' 3 4 4' 5' 6-Heptachlorobiphenyl (BZ 183)
GC/ECD	8082/8082A	2 2' 3 4 4' 5'-Hexachlorobiphenyl (BZ 138)
GC/ECD	8082/8082A	2 2' 3 4 4' 6 6'-Heptachlorobiphenyl (BZ 184)
GC/ECD	8082/8082A	2 2' 3 4' 5 5' 6-Heptachlorobiphenyl (BZ 187)
GC/ECD	8082/8082A	2 2' 3 4 5'-Pentachlorobiphenyl (BZ 87)
GC/ECD	8082/8082A	2 2' 3 5'-Tetrachlorobiphenyl (BZ 44)
GC/ECD	8082/8082A	2 2' 4 4' 5 5'-Hexachlorobiphenyl (BZ 153)
GC/ECD	8082/8082A	2 2' 4 5 5'-Pentachlorobiphenyl (BZ 101)
GC/ECD	8082/8082A	2 2' 4' 5-Tetrachlorobiphenyl (BZ 49)
GC/ECD	8082/8082A	2 2' 5 5'-Tetrachlorobiphenyl (BZ 52)
GC/ECD	8082/8082A	2 2' 5-Trichlorobiphenyl (BZ 18)
GC/ECD	8082/8082A	2 3 3' 4 4' 5-Hexachlorobiphenyl (BZ 156)
GC/ECD	8082/8082A	2 3 3' 4 4' 5'-Hexachlorobiphenyl (BZ 157)
GC/ECD	8082/8082A	2 3 3' 4 4'-Pentachlorobiphenyl (BZ 105)
GC/ECD	8082/8082A	2 3 3' 4 4' 5 5'-Heptachlorobiphenyl (BZ 189)
GC/ECD	8082/8082A	2 3' 4 4' 5 5'-Hexachlorobiphenyl (BZ 167)
GC/ECD	8082/8082A	2 3' 4 4' 5-Pentachlorobiphenyl (BZ 118)
GC/ECD	8082/8082A	2 3' 4 4'5-Pentachlorobiphenyl (BZ 123)
GC/ECD	8082/8082A	2 3' 4 4'-Tetrachlorobiphenyl (BZ 66)
GC/ECD	8082/8082A	2 3' 4 4' 5-Pentachlorobiphenyl (BZ 114)
GC/ECD	8082/8082A	2 4 4'-Trichlorobiphenyl (BZ 28)
GC/ECD	8082/8082A	2 4'-Dichlorobiphenyl (BZ 8)
GC/ECD	8082/8082A	3 3' 4 4' 5 5'-Hexachlorobiphenyl (BZ 169)
GC/ECD	8082/8082A	3 3' 4 4' 5-Pentachlorobiphenyl (BZ 126)
GC/ECD	8082/8082A	3 3' 4 4'-Tetrachlorobiphenyl (BZ 77)

Solid and Chemical Waste		
Technology	**Method**	**Analyte**
GC/ECD	8082/8082A	3 4 4' 5-Tetrachlorobiphenyl (BZ 81)
GC/ECD	8082/8082A	Decachlorobiphenyl (BZ 209)
GC/ECD	8151A	2 4 5-T
GC/ECD	8151A	2 4-D
GC/ECD	8151A	2 4-DB
GC/ECD	8151A	Dalapon
GC/ECD	8151A	Dicamba
GC/ECD	8151A	Dichloroprop
GC/ECD	8151A	DInoseb
GC/ECD	8151A	MCPA
GC/ECD	8151A	MCPP
GC/ECD	8151A	Pentachlorophenol
GC/ECD	8151A	Silvex (2 4 5-TP)
GC/FID	8015B,C	Diesel range organics (DRO)
GC/FID	8015B,C	Gasoline range organics (GRO)
GC/FID	8011	EDB
GC/FID	8011	1 2-Dibromo-3-chloropropane
GC/MS	8260B,C	1 1 1 2-Tetrachloroethane
GC/MS	8260B,C / SOM01.2	1 1 1-Trichloroethane
GC/MS	8260B,C / SOM01.2	1 1 2 2-Tetrachloroethane
GC/MS	SOM01.2	1 1 2-Trichloro-1 2 2-trifluoroethane
GC/MS	8260B,C / SOM01.2	1 1 2-Trichloroethane
GC/MS	8260B,C / SOM01.2	1 1-Dichloroethane
GC/MS	8260B,C / SOM01.2	1 1-Dichloroethylene
GC/MS	8260B,C	1 1-Dichloropropene
GC/MS	8260B,C / SOM01.2	1 2 3-Trichlorobenzene
GC/MS	8260B,C	1 2 3-Trichloropropane
GC/MS	8260B,C / SOM01.2	1 2 4-Trichlorobenzene
GC/MS	8260B,C	1 2 4-Trimethylbenzene
GC/MS	8260B,C / SOM01.2	1 2-Dibromo-3-chloropropane
GC/MS	8260B,C / SOM01.2	1 2-Dichlorobenzene
GC/MS	8260B,C / SOM01.2	1 2-Dichloroethane
GC/MS	8260B,C / SOM01.2	1 2-Dichloropropane
GC/MS	8260B,C	1 3 5-Trimethylbenzene
GC/MS	8260B,C / SOM01.2	1 3-Dichlorobenzene
GC/MS	8260B,C	1 3-Dichloropropane
GC/MS	8260B,C / SOM01.2	1 4-Dichlorobenzene
GC/MS	8260B,C / SOM01.2	1 4-Dioxane
GC/MS	8260B,C	2 2-Dichloropropane
GC/MS	8260B,C / SOM01.2	2-Butanone
GC/MS	8260B,C	2-Chloroethyl vinyl ether
GC/MS	8260B,C	2-Chlorotoluene
GC/MS	8260B,C / SOM01.2	2-Hexanone
GC/MS	8260B,C	4-Chlorotoluene
GC/MS	8260B,C / SOM01.2	4-Methyl-2-pentanone

Solid and Chemical Waste		
Technology	**Method**	**Analyte**
GC/MS	8260B,C / SOM01.2	Acetone
GC/MS	8260B,C	Acetonitrile
GC/MS	8260B,C	Acrolein
GC/MS	8260B,C	Acrylonitrile
GC/MS	8260B,C	Allyl chloride
GC/MS	8260B,C / SOM01.2	Benzene
GC/MS	8260B,C	Bromobenzene
GC/MS	8260B,C / SOM01.2	Bromochloromethane
GC/MS	8260B,C / SOM01.2	Bromodichloromethane
GC/MS	8260B,C / SOM01.2	Bromoform
GC/MS	8260B,C / SOM01.2	Carbon disulfide
GC/MS	8260B,C / SOM01.2	Carbon tetrachloride
GC/MS	8260B,C / SOM01.2	Chlorobenzene
GC/MS	8260B,C / SOM01.2	Chloroethane
GC/MS	8260B,C / SOM01.2	Chloroform
GC/MS	8260B,C	Chloroprene
GC/MS	8260B,C / SOM01.2	cis-1 2-Dichloroethene
GC/MS	8260B,C / SOM01.2	cis-1 3-Dichloropropene
GC/MS	SOM01.2	Cyclohexane
GC/MS	8260B,C / SOM01.2	Dibromochloromethane
GC/MS	8260B,C	Dibromomethane
GC/MS	624 / 8260B,C / SOM01.2	Dichlorodifluoromethane
GC/MS	8260B,C	Diethyl ether
GC/MS	8260B,C	Di-isopropylether
GC/MS	8260B,C / SOM01.2	EDB
GC/MS	8260B,C	Ethyl methacrylate
GC/MS	8260B,C / SOM01.2	Ethylbenzene
GC/MS	8260B,C	Ethyl-t-butylether
GC/MS	8260B,C	Hexachlorobutadiene
GC/MS	8260B,C	Iodomethane
GC/MS	8260B,C	Isobutyl alcohol
GC/MS	8260B,C / SOM01.2	Isopropyl benzene
GC/MS	SOM01.2	m p-xylenes
GC/MS	8260B,C	Methacrylonitrile
GC/MS	SOM01.2	Methyl acetate
GC/MS	8260B,C / SOM01.2	Methyl bromide (Bromomethane)
GC/MS	8260B,C / SOM01.2	Methyl chloride (Chloromethane)
GC/MS	8260B,C	Methyl methacrylate
GC/MS	8260B,C / SOM01.2	Methyl tert-butyl ether
GC/MS	SOM01.2	Methylcyclohexane
GC/MS	8260B,C / SOM01.2	Methylene chloride
GC/MS	8260B,C	Naphthalene
GC/MS	8260B,C	n-Butylbenzene
GC/MS	8260B,C	n-proplybenzene
GC/MS	8260B,C	o-Xylene

Solid and Chemical Waste		
Technology	**Method**	**Analyte**
GC/MS	8260B,C	p-Isopropyltoluene
GC/MS	8260B,C	Propionitrile
GC/MS	8260B,C	sec-butylbenzene
GC/MS	8260B,C / SOM01.2	Styrene
GC/MS	8260B,C	t-Amylmethylether
GC/MS	8260B,C	tert-Butyl alcohol
GC/MS	8260B,C	tert-Butylbenzene
GC/MS	8260B,C / SOM01.2	Tetrachloroethylene (Perchloroethylene)
GC/MS	8260B,C	Tetrahydrofuran
GC/MS	8260B,C / SOM01.2	Toluene
GC/MS	8260B,C / SOM01.2	trans-1 2-Dichloroethylene
GC/MS	8260B,C / SOM01.2	trans-1 3-Dichloropropylene
GC/MS	8260B,C	Trans-1 4-Dichloro-2-butuene
GC/MS	8260B,C / SOM01.2	Trichloroethene (Trichloroethylene)
GC/MS	8260B,C / SOM01.2	Trichlorofluoromethane
GC/MS	8260B,C	Vinyl acetate
GC/MS	8260B,C / SOM01.2	Vinyl chloride
GC/MS	8260B,C	Xylene
GC/MS	8270C,D	1-Naphthylamine
GC/MS	8270C,D	2-Acetylaminofluorene
GC/MS	8270C,D / SOM01.2	2-Chloronaphthalene
GC/MS	8270C,D / SOM01.2	2-Chlorophenol
GC/MS	8270C,D / SOM01.2	2-Methylnaphthalene
GC/MS	8270C,D / SOM01.2	2-Methylphenol
GC/MS	8270C,D	2-Naphthylamine
GC/MS	8270C,D	2-Nitroaniline
GC/MS	8270C,D / SOM01.2	2-Nitrophenol
GC/MS	8270C,D	2-Picoline
GC/MS	8270C,D	3-Methylcholanthrene
GC/MS	8270C,D / SOM01.2	3-Nitroaniline
GC/MS	8270C,D	4-Aminobiphenyl
GC/MS	8270C,D / SOM01.2	4-Bromophenyl phenyl ether
GC/MS	8270C,D / SOM01.2	4-Chloro-3-methylphenol
GC/MS	8270C,D / SOM01.2	4-Chloroaniline
GC/MS	8270C,D / SOM01.2	4-Chlorophenyl phenylether
GC/MS	8270C,D	4-Dimethyl aminoazobenzene
GC/MS	8270C,D / SOM01.2	4-Methylphenol
GC/MS	8270C,D / SOM01.2	4-Nitroaniline
GC/MS	8270C,D / SOM01.2	4-Nitrophenol
GC/MS	8270C,D	5-Nitro-o-toluidine
GC/MS	8270C,D	a a-Dimethylphenethylamine
GC/MS	8270C,D / SOM01.2	Acenaphthene
GC/MS	8270C,D / SOM01.2	Acenaphthylene
GC/MS	8270C,D / SOM01.2	Acetophenone
GC/MS	8270C,D	Aniline

Solid and Chemical Waste		
Technology	**Method**	**Analyte**
GC/MS	8270C,D / SOM01.2	Anthracene
GC/MS	8270C,D	Aramite
GC/MS	8270C,D / SOM01.2	Atrazine
GC/MS	SOM01.2	Benzaldehyde
GC/MS	8270C,D	Benzidine
GC/MS	8270C,D / SOM01.2	Benzo(a)anthracene
GC/MS	8270C,D / SOM01.2	Benzo(a)pyrene
GC/MS	8270C,D / SOM01.2	Benzo(b)fluoranthene
GC/MS	8270C,D / SOM01.2	Benzo(g h i)perylene
GC/MS	8270C,D / SOM01.2	Benzo(k)fluoranthene
GC/MS	8270C,D	Benzoic Acid
GC/MS	8270C,D	Benzyl alcohol
GC/MS	8270C,D / SOM01.2	Biphenyl
GC/MS	8270C,D / SOM01.2	bis(2-Chloroethoxy)methane
GC/MS	8270C,D / SOM01.2	bis(2-Chloroethyl) ether
GC/MS	8270C,D / SOM01.2	bis(2-Ethylhexyl) phthalate (DEHP)
GC/MS	8270C,D / SOM01.2	Butyl benzyl phthalate
GC/MS	SOM01.2	Caprolactam
GC/MS	8270C,D / SOM01.2	Carbazole
GC/MS	8270C,D	Chlorobenzilate
GC/MS	8270C,D / SOM01.2	Chrysene
GC/MS	8270C,D	Diallate
GC/MS	8270C,D / SOM01.2	Dibenz(a h)anthracene
GC/MS	8270C,D / SOM01.2	Dibenzofuran
GC/MS	8270C,D / SOM01.2	Diethyl phthalate
GC/MS	8270C,D	Dimethoate
GC/MS	8270C,D / SOM01.2	Dimethyl phthalate
GC/MS	8270C,D / SOM01.2	Di-n-butyl phthalate
GC/MS	8270C,D / SOM01.2	Di-n-octyl phthalate
GC/MS	8270C,D	Ethyl methanesulfonate
GC/MS	8270C,D	Famfur
GC/MS	8270C,D / SOM01.2	Fluoranthene
GC/MS	8270C,D / SOM01.2	Fluorene
GC/MS	8270C,D / SOM01.2	Hexachlorobenzene
GC/MS	8270C,D / SOM01.2	Hexachlorobutadiene
GC/MS	8270C,D / SOM01.2	Hexachlorocyclopentadiene
GC/MS	8270C,D / SOM01.2	Hexachloroethane
GC/MS	8270C,D	Hexachloropropene
GC/MS	8270C,D	Isodrin
GC/MS	8270C,D / SOM01.2	Isophorone
GC/MS	8270C,D	Isosafrole
GC/MS	8270C,D	Methapyriline
GC/MS	8270C,D	Methyl methanesulfonate
GC/MS	8270C,D	Methyl parathion
GC/MS	8270C,D / SOM01.2	Naphthalene

Solid and Chemical Waste		
Technology	**Method**	**Analyte**
GC/MS	8270C,D / SOM01.2	Nitrobenzene
GC/MS	8270C,D	Nitroquinoline-1-oxide
GC/MS	8270C,D	n-Nitrosodiethylamine
GC/MS	8270C,D / SOM01.2	n-Nitrosodimethylamine
GC/MS	8270C,D	n-Nitroso-di-n-butylamine
GC/MS	8270C,D / SOM01.2	n-Nitrosodi-n-propylamine
GC/MS	8270C,D / SOM01.2	n-Nitrosodiphenylamine
GC/MS	8270C,D	n-Nitrosomethylethylamine
GC/MS	8270C,D	n-Nitrosomorpholine
GC/MS	8270C,D	n-Nitrosopiperidine
GC/MS	8270C,D	n-Nitrosopyrrolidine
GC/MS	8270C,D	o o o-Triethyl phosphorothioate
GC/MS	8270C,D	o-Toluidine
GC/MS	8270C,D	Pentachlorobenzene
GC/MS	8270C,D	Pentachloronitrobenzene
GC/MS	8270C,D/ SOM01.2	Pentachlorophenol
GC/MS	8270C,D	Phenacetin
GC/MS	8270C,D / SOM01.2	Phenanthrene
GC/MS	8270C,D / SOM01.2	Phenol
GC/MS	8270C,D	Phorate
GC/MS	8270C,D	Pronamide
GC/MS	8270C,D / SOM01.2	Pyrene
GC/MS	8270C,D	Pyrididne
GC/MS	8270C,D	Safrole
GC/MS	8270C,D	Thionazin
GC/MS	8270C,D / SOM01.2	Indeno(1 2 3-cd)pyrene
GC/MS	8270C,D / SOM01.2	1 2 4-Trichlorobenzene
GC/MS	8270C,D	1 3 5-Trinitrobenzene
GC/MS	8270C,D / SOM01.2	1 2 4 5-Tetrachlorobenzene
GC/MS	8270C,D / SOM01.2	2 4 5-Trochlorophenol
GC/MS	8270C,D / SOM01.2	2 4 6-Trichlorophenol
GC/MS	8270C,D / SOM01.2	2 3 4 6-Tetrachlorophenol
GC/MS	8270C,D / SOM01.2	1 2-Dichlorobenzene
GC/MS	8270C,D	1 2-Diphenylhydrazine
GC/MS	8270C,D / SOM01.2	1 3-Dichlorobenzene
GC/MS	8270C,D	1 3-Dinitrobenzene
GC/MS	8270C,D / SOM01.2	1 4-Dichlorobenzene
GC/MS	8270C,D	1 4-Dioxane
GC/MS	8270C,D	1 4-Naphthoquinone
GC/MS	8270C,D	1 4-Phenylenediamine
GC/MS	8270C,D / SOM01.2	bis(2-Chloroisopropyl) ether (2 2`-Oxybis(1-chloropropane))
GC/MS	8270C,D / SOM01.2	2 4-Dichlorophenol
GC/MS	8270C,D / SOM01.2	2 4-Dimethylphenol
GC/MS	8270C,D / SOM01.2	2 4-Dinitrophenol

Solid and Chemical Waste		
Technology	**Method**	**Analyte**
GC/MS	8270C,D / SOM01.2	2 4-Dinitrotoluene (2 4-DNT)
GC/MS	8270C,D	2 6-Dichlorophenol
GC/MS	8270C,D / SOM01.2	2 6-Dinitrotoluene (2 6-DNT)
GC/MS	8270C,D / SOM01.2	3 3`-Dichlorobenzidine
GC/MS	8270C,D	3 3'-Dimethylbenzidine
GC/MS	8270C,D / SOM01.2	2-Methyl-4 6-dinitrophenol
GC/MS	8270C,D	7,12-Dimethylphenethylamine
HPLC	8330/8330A/8330B (Analysis Only)	1 3 5-Trinitrobenzene
HPLC	8330/8330A/8330B (Analysis Only)	1 3-Dinitrobenzene
HPLC	8330/8330A/8330B (Analysis Only)	2 4 6-Trinitrotoluene
HPLC	8330/8330A/8330B (Analysis Only)	2 4-Dinitrotoluene
HPLC	8330/8330A/8330B (Analysis Only)	2 6-Dinitrotoluene
HPLC	8330/8330A/8330B (Analysis Only)	2-Amino-4 6 -dinitrotoluene
HPLC	8330/8330A/8330B (Analysis Only)	2-Nitrotoluene
HPLC	8330/8330A/8330B (Analysis Only)	3-Nitrotoluene
HPLC	8330/8330A/8330B (Analysis Only)	4-Amino-2,3-dinitrotoluene
HPLC	8330/8330A/8330B (Analysis Only)	4-Nitrotoluene
HPLC	8330/8330A/8330B (Analysis Only)	Hexahydr-1 3 5-trinitro-1 3 5-triazine (RDX)
HPLC	8330/8330A/8330B (Analysis Only)	Nitrobenzene
HPLC	8330/8330A/8330B (Analysis Only)	Nitroglycerin
HPLC	8330/8330A/8330B (Analysis Only)	Octahydro-1 3 5 7-tetrazocine (HMX)
HPLC	8330/8330A/8330B (Analysis Only)	Tetryl
CVAA	7471B/ ILM05.3	Mercury
CVAF	1631E	Low Level Mercury
ICP	6010B,C /ILM05.3	Aluminum
ICP	6010B,C /ILM05.3	Antimony
ICP	6010B,C /ILM05.3	Arsenic
ICP	6010B,C /ILM05.3	Barium
ICP	6010B,C /ILM05.3	Beryllium
ICP	6010B,C	Boron
ICP	6010B,C /ILM05.3	Cadmium

Solid and Chemical Waste		
Technology	**Method**	**Analyte**
ICP	6010B,C /ILM05.3	Calcium
ICP	6010B,C /ILM05.3	Chromium
ICP	6010B,C /ILM05.3	Cobalt
ICP	6010B,C /ILM05.3	Copper
ICP	6010B,C /ILM05.3	Iron
ICP	6010B,C /ILM05.3	Lead
ICP	6010B,C /ILM05.3	Magnesium
ICP	6010B,C /ILM05.3	Manganese
ICP	6010B,C	Molybdenum
ICP	6010B,C /ILM05.3	Nickel
ICP	6010B,C /ILM05.3	Potassium
ICP	6010B,C /ILM05.3	Selenium
ICP	200.7	Silicon
ICP	6010B,C /ILM05.3	Silver
ICP	6010B,C /ILM05.3	Sodium
ICP	6010B,C	Strontium
ICP	6010B,C /ILM05.3	Thallium
ICP	6010B,C	Tin
ICP	6010B,C	Titanium
ICP	6010B,C /ILM05.3	Vanadium
ICP	6010B,C /ILM05.3	Zinc
ICP/MS	6020/6020A / ILM05.3	Aluminum
ICP/MS	6020/6020A / ILM05.3	Antimony
ICP/MS	6020/6020A / ILM05.3	Arsenic
ICP/MS	6020/6020A / ILM05.3	Barium
ICP/MS	6020/6020A / ILM05.3	Beryllium
ICP/MS	6020/6020A	Boron
ICP/MS	6020/6020A / ILM05.3	Cadmium
ICP/MS	6020/6020A	Calcium
ICP/MS	6020/6020A / ILM05.3	Chromium
ICP/MS	6020/6020A / ILM05.3	Cobalt
ICP/MS	6020/6020A / ILM05.3	Copper
ICP/MS	6020/6020A	Iron
ICP/MS	6020/6020A / ILM05.3	Lead
ICP/MS	6020/6020A / ILM05.3	Magnesium
ICP/MS	6020/6020A	Manganese
ICP/MS	6020/6020A	Molybdenum
ICP/MS	6020/6020A / ILM05.3	Nickel
ICP/MS	6020/6020A	Potassium
ICP/MS	6020/6020A / ILM05.3	Selenium
ICP/MS	6020/6020A / ILM05.3	Silver
ICP/MS	6020/6020A	Sodium
ICP/MS	6020/6020A	Strontium
ICP/MS	6020/6020A / ILM05.3	Thallium
ICP/MS	6020/6020A	Tin

Solid and Chemical Waste		
Technology	**Method**	**Analyte**
ICP/MS	6020/6020A	Titanium
ICP/MS	6020/6020A / ILM05.3	Vanadium
ICP/MS	6020/6020A / ILM05.3	Zinc
IC	9056/9056A	Chloride
IC	9056/9056A	Fluoride
IC	9056/9056A	Nitrate as N
IC	9056/9056A	Nitrite as N
IC	9056/9056A	Sulfate
Gravimetric	9070A / 9071B	Oil and Grease
Physical	1010A	Ignitability
Physical	9045D	pH
Titration	Chap 7.3.4	Reactive Sulfide
TOC	Lloyd Kahn	Total organic carbon
TOC	9060A / 5310B	Total organic carbon
Turbidimetric	9038 / ASTM 516-02	Sulfate
UV/VIS	350.1 / 4500NH3 H	Ammonia as N
UV/VIS	9251 / 4500Cl E	Chloride
UV/VIS	Chap. 7.3.4	Reactive Cyanide
UV/VIS	376.3	AVS-SEM
UV/VIS	3500Fe D	Ferrous Iron
Cleanup Methods	3630C	Silica Gel
UV/VIS	7196	Chromium VI
UV/VIS	7196A	Chromium VI
UV/VIS	9012B / ILM05.3	Total cyanide
Preparation	**Method**	**Type**
Preparation	1311	Toxicity Characteristic Leaching Procedure
Preparation	1312	Synthetic Precipitation Leaching Procedure
Cleanup Methods	3660B	Sulfur
Cleanup Methods	3620C	Florsil
Cleanup Methods	3630C	Silica Gel
Cleanup Methods	3640A	GPC
Organic Preparation	3540C	Soxhlet Extraction
Organic Preparation	3545A	Pressurized Fluid Extraction
Organic Preparation	3550C	Sonication
Inorganics Preparation	3050B	Hotblock
Inorganics Preparation	3060A	Alkaline Digestion
Volatile Organics Preparation	5035/5035A	Closed System Purge and Trap

Notes:

1) This laboratory offers commercial testing service.

Approved By: _____ Date: November 4, 2009

R. Douglas Leonard
Chief Technical Officer

Issued: 11/04/09

Target List	EPA Method	Soil Accuracy % Recovery	Aqueous Accuracy % Recovery	Soil Precision RPD (1)	Aqueous Precision RPD (1)	Soil PQLs	Water PQLs	Soil MDLs	Water MDLs
Polychorinated Biphenyls						**ug/Kg**	**ug/L**	**ug/Kg**	**ug/L**
Aroclor 1016	8082	**53-123**	**65-112**	0-50 (nominal)	0-30 (nominal)	17	0.50	6.0	0.15
Aroclor 1221	8082	---	---	---	---	17	0.50	7.9	0.20
Aroclor 1232	8082	---	---	---	---	17	0.50	9.3	0.089
Aroclor 1242	8082	---	---	---	---	17	0.50	5.8	0.18
Aroclor 1248	8082	---	---	---	---	17	0.50	6.1	0.200
Aroclor 1254	8082	---	---	---	---	17	0.50	4.7	0.082
Aroclor 1260	8082	**58-120**	**62-104**	0-50 (nominal)	0-30 (nominal)	17	0.50	6.0	0.17
Surrogates:									
Tetrachloro-m-xylene	8082	56-115	62-111	---	---	---	---	---	---
Decachlorobiphenyl	8082	59-124	44-135	---	---	---	---	---	---
Volatile Organic Compounds						**ug/Kg**	**ug/L**	**ug/Kg**	**ug/L**
Dichlorodifluoromethane	8260B	45-167	29-164	0-30 (nominal)	0-20 (nominal)	10	2.0	0.92	0.24
Chloromethane	8260B	69-127	59-123	0-30 (nominal)	0-20 (nominal)	10	2.0	1.4	0.36
Vinyl Chloride	8260B	73-134	64-131	0-30 (nominal)	0-20 (nominal)	10	2.0	0.87	0.25
Bromomethane	8260B	64-136	57-135	0-30 (nominal)	0-20 (nominal)	10	2.0	1.1	0.49
Chloroethane	8260B	71-127	53-157	0-30 (nominal)	0-20 (nominal)	10	2.0	1.3	0.55
Trichlorofluoromethane	8260B	73-145	70-149	0-30 (nominal)	0-20 (nominal)	10	2.0	0.91	0.24
Diethyl Ether	8260B	76-135	78-124	0-30 (nominal)	0-20 (nominal)	5.0	1.0	0.80	0.40
Tertiary-butyl alcohol	8260B	62-148	11-151	0-30 (nominal)	0-20 (nominal)	25	5.0	11	2.47
1,1-Dichloroethene	8260B	71-137	88-127	0-30 (nominal)	0-20 (nominal)	5.0	1.0	0.93	0.35
Carbon Disulfide	8260B	69-138	71-129	0-30 (nominal)	0-20 (nominal)	5.0	1.0	0.78	0.25
1,1,2-Trichloro-1,2,2-triflurorethane (F	8260B	67-135	73-126	0-30 (nominal)	0-20 (nominal)	5.0	1.0	0.90	0.31
Iodomethane	8260B	51-150	54-155	0-30 (nominal)	0-20 (nominal)	25	5.0	2.4	0.31
Acrolein	8260B	48-175	62-135	0-30 (nominal)	0-20 (nominal)	50	10	6.6	2.82
Methylene Chloride	8260B	56-152	72-129	0-30 (nominal)	0-20 (nominal)	25	5.0	7.9	1.13
Acetone	8260B	76-213	62-172	0-30 (nominal)	0-20 (nominal)	25	5.0	5.1	2.21
Isobutyl alcohol	8260B	72-142	16-147	0-30 (nominal)	0-20 (nominal)	100	20	16	7.8
trans-1,2-Dichloroethene	8260B	67-133	78-125	0-30 (nominal)	0-20 (nominal)	5.0	1.0	0.71	0.25
Allyl chloride	8260B	69-135	78-121	0-30 (nominal)	0-20 (nominal)	5.0	1.0	0.82	0.33
Methyl tert-butyl ether	8260B	81-125	81-125	0-30 (nominal)	0-20 (nominal)	5.0	1.0	1.1	0.36
Acetonitrile	8260B	63-143	61-125	0-30 (nominal)	0-20 (nominal)	125	25	51	7.4
Di-isopropyl ether	8260B	68-131	81-123	0-30 (nominal)	0-20 (nominal)	5.0	1.0	2.1	0.21
Chloroprene	8260B	67-130	75-128	0-30 (nominal)	0-20 (nominal)	5.0	1.0	0.98	0.27
Propionitrile	8260B	73-133	75-118	0-30 (nominal)	0-20 (nominal)	50	10	7.5	4.42
Methacrylonitrile	8260B	79-129	78-123	0-30 (nominal)	0-20 (nominal)	50	10	6.0	2.05
1,1-Dichloroethane	8260B	75-126	76-130	0-30 (nominal)	0-20 (nominal)	5.0	1.0	1.7	0.21

Target List	EPA Method	Soil Accuracy % Recovery	Aqueous Accuracy % Recovery	Soil Precision RPD (1)	Aqueous Precision RPD (1)	Soil PQLs	Water PQLs	Soil MDLs	Water MDLs
Acrylonitrile	8260B	69-139	76-120	0-30 (nominal)	0-20 (nominal)	25	5.0	13	1.47
Ethyl tertiary-butyl ether	8260B	83-122	85-119	0-30 (nominal)	0-20 (nominal)	5.0	1.0	0.59	0.23
Vinyl Acetate	8260B	77-119	56-129	0-30 (nominal)	0-20 (nominal)	5.0	1.0	0.94	0.40
cis-1,2-Dichloroethene	8260B	82-123	85-123	0-30 (nominal)	0-20 (nominal)	5.0	1.0	0.91	0.21
1,2-Dichloroethene (Total)	8260B	82-120	84-121	0-30 (nominal)	0-20 (nominal)	10	2.0	0.71	0.21
Methyl methacrylate	8260B	82-130	79-121	0-30 (nominal)	0-20 (nominal)	5.0	1.0	0.72	0.29
2,2-Dichloropropane	8260B	78-124	70-132	0-30 (nominal)	0-20 (nominal)	5.0	1.0	0.50	0.25
Bromochloromethane	8260B	84-115	85-117	0-30 (nominal)	0-20 (nominal)	5.0	1.0	0.91	0.21
Chloroform	8260B	83-118	78-128	0-30 (nominal)	0-20 (nominal)	5.0	1.0	0.35	0.32
Carbon Tetrachloride	8260B	78-124	87-126	0-30 (nominal)	0-20 (nominal)	5.0	1.0	1.3	0.22
Tetrahydrofuran	8260B	78-137	74-123	0-30 (nominal)	0-20 (nominal)	50	10	4.5	1.71
1,1,1-Trichloroethane	8260B	80-120	77-129	0-30 (nominal)	0-20 (nominal)	5.0	1.0	0.42	0.20
1,1-Dichloropropene	8260B	81-119	87-118	0-30 (nominal)	0-20 (nominal)	5.0	1.0	0.91	0.21
2-Butanone (MEK)	8260B	78-148	71-132	0-30 (nominal)	0-20 (nominal)	25	5	5.9	1.31
Benzene	8260B	82-113	86-116	0-30 (nominal)	0-20 (nominal)	5.0	1.0	0.92	0.26
Cyclohexane	8260B	75-128	71-133	0-30 (nominal)	0-20 (nominal)	5.0	1.0	1.4	0.31
Ethyl methacrylate	8260B	78-128	80-125	0-30 (nominal)	0-20 (nominal)	5.0	1.0	1.1	0.37
Tertiary-amyl methyl ether	8260B	80-123	80-121	0-30 (nominal)	0-20 (nominal)	5.0	1.0	0.39	0.21
1,2-Dichloroethane	8260B	83-121	81-125	0-30 (nominal)	0-20 (nominal)	5.0	1.0	1.0	0.20
Trichloroethene	8260B	83-113	79-121	0-30 (nominal)	0-20 (nominal)	5.0	1.0	0.59	0.28
Dibromomethane	8260B	85-118	85-117	0-30 (nominal)	0-20 (nominal)	5.0	1.0	0.51	0.46
1,2-Dichloropropane	8260B	84-115	84-118	0-30 (nominal)	0-20 (nominal)	5.0	1.0	1.4	0.25
Bromodichloromethane	8260B	82-118	85-122	0-30 (nominal)	0-20 (nominal)	5.0	1.0	0.60	0.33
cis-1,3-Dichloropropene	8260B	80-115	83-119	0-30 (nominal)	0-20 (nominal)	5.0	1.0	0.72	0.19
1,4-Dioxane	8260B	69-137	10-149	0-30 (nominal)	0-20 (nominal)	500	100	33.0	8.8
2-Chloroethylvinylether	8260B	16-150	39-135	0-30 (nominal)	0-20 (nominal)	25	5.0	2.3	0.69
Toluene	8260B	80-113	84-118	0-30 (nominal)	0-20 (nominal)	5.0	1.0	1.4	0.27
4-methyl-2-pentanone	8260B	75-137	83-122	0-30 (nominal)	0-20 (nominal)	25.0	5.0	5.9	1.32
Tetrachloroethene	8260B	73-122	47-155	0-30 (nominal)	0-20 (nominal)	5.0	1.0	1.2	0.40
trans-1,3-Dichloropropene	8260B	87-136	85-135	0-30 (nominal)	0-20 (nominal)	5.0	1.0	0.86	0.20
1,1,2-Trichloroethane	8260B	78-117	84-115	0-30 (nominal)	0-20 (nominal)	5.0	1.0	0.97	0.33
Dibromochloromethane	8260B	80-120	85-119	0-30 (nominal)	0-20 (nominal)	5.0	1.0	1.0	0.30
1,3-Dichloropropane	8260B	80-114	80-119	0-30 (nominal)	0-20 (nominal)	5.0	1.0	0.94	0.22
1,2-Dibromoethane	8260B	81-119	84-116	0-30 (nominal)	0-20 (nominal)	5.0	1.0	1.2	0.22
2-Hexanone	8260B	72-149	80-124	0-30 (nominal)	0-20 (nominal)	25	5	4.8	1.70
Chlorobenzene	8260B	85-111	89-113	0-30 (nominal)	0-20 (nominal)	5.0	1.0	0.51	0.22
1-Chlorohexane	8260B	70-122	73-119	0-30 (nominal)	0-20 (nominal)	5.0	1.0	1.5	0.25

Target List	EPA Method	Soil Accuracy % Recovery	Aqueous Accuracy % Recovery	Soil Precision RPD (1)	Aqueous Precision RPD (1)	Soil PQLs	Water PQLs	Soil MDLs	Water MDLs
Ethylbenzene	8260B	81-112	88-113	0-30 (nominal)	0-20 (nominal)	5.0	1.0	0.65	0.21
1,1,1,2-Tetrachloroethane	8260B	83-114	88-118	0-30 (nominal)	0-20 (nominal)	5.0	1.0	0.70	0.19
Xylenes (Total)	8260B	81-114	89-116	0-30 (nominal)	0-20 (nominal)	15	3.0	1.3	0.25
m+p-Xylene	8260B	80-115	88-116	0-30 (nominal)	0-20 (nominal)	10	2.0	1.7	0.59
o-Xylene	8260B	82-115	90-116	0-30 (nominal)	0-20 (nominal)	5.0	1.0	1.3	0.25
Styrene	8260B	84-112	88-117	0-30 (nominal)	0-20 (nominal)	5.0	1.0	0.51	0.23
Bromoform	8260B	76-126	86-117	0-30 (nominal)	0-20 (nominal)	5.0	1.0	0.70	0.23
Isopropylbenzene	8260B	89-136	96-136	0-30 (nominal)	0-20 (nominal)	5.0	1.0	0.92	0.23
cis-1,4-Dichloro-2-Butene	8260B	73-139	59-136	0-30 (nominal)	0-20 (nominal)	5.0	1.0	1.1	0.47
trans-1,4-Dichloro-2-Butene	8260B	65-137	63-132	0-30 (nominal)	0-20 (nominal)	5.0	1.0	1.3	0.39
Bromobenzene	8260B	78-118	84-113	0-30 (nominal)	0-20 (nominal)	5.0	1.0	0.74	0.24
n-Propylbenzene	8260B	77-121	83-121	0-30 (nominal)	0-20 (nominal)	5.0	1.0	0.83	0.26
1,1,2,2-Tetrachloroethane	8260B	78-122	79-121	0-30 (nominal)	0-20 (nominal)	5.0	1.0	0.84	0.38
1,3,5-Trimethylbenzene	8260B	79-116	80-123	0-30 (nominal)	0-20 (nominal)	5.0	1.0	0.67	0.20
2-Chlorotoluene	8260B	78-118	81-120	0-30 (nominal)	0-20 (nominal)	5.0	1.0	1.1	0.20
1,2,3-Trichloropropane	8260B	81-118	77-120	0-30 (nominal)	0-20 (nominal)	5.0	1.0	1.2	0.19
4-Chlorotoluene	8260B	77-121	81-122	0-30 (nominal)	0-20 (nominal)	5.0	1.0	0.49	0.26
tert-Butylbenzene	8260B	79-118	84-121	0-30 (nominal)	0-20 (nominal)	5.0	1.0	0.90	0.31
Pentachloroethane	8260B	78-127	19-186	0-30 (nominal)	0-20 (nominal)	5.0	1.0	1.9	0.98
1,2,4-Trimethylbenzene	8260B	76-115	83-118	0-30 (nominal)	0-20 (nominal)	5.0	1.0	0.87	0.19
p-Isopropyltoluene	8260B	80-124	88-121	0-30 (nominal)	0-20 (nominal)	5.0	1.0	0.76	0.25
1,3-Dichlorobenzene	8260B	79-119	86-110	0-30 (nominal)	0-20 (nominal)	5.0	1.0	0.62	0.26
1,4-Dichlorobenzene	8260B	80-117	86-111	0-30 (nominal)	0-20 (nominal)	5.0	1.0	0.44	0.24
n-Butylbenzene	8260B	70-124	78-121	0-30 (nominal)	0-20 (nominal)	5.0	1.0	0.92	0.23
Sec-Butylbenzene	8260B	75-122	82-122	0-30 (nominal)	0-20 (nominal)	5.0	1.0	0.91	0.21
1,2-Dichlorobenzene	8260B	76-118	86-112	0-30 (nominal)	0-20 (nominal)	5.0	1.0	0.78	0.15
1,2-Dibromo-3-Chloropropane	8260B	66-132	67-124	0-30 (nominal)	0-20 (nominal)	5.0	1.0	1.5	0.50
1,3,5-Trichlorobenzene	8260B	64-133	77-120	0-30 (nominal)	0-20 (nominal)	5.0	1.0	0.87	0.24
Hexachlorobutadiene	8260B	69-120	73-113	0-30 (nominal)	0-20 (nominal)	5.0	1.0	0.74	0.52
1,2,4-Trichlorobenzene	8260B	61-135	76-126	0-30 (nominal)	0-20 (nominal)	5.0	1.0	0.79	0.37
1,2,3-Trimethylbenzene	8260B	82-116	85-119	0-30 (nominal)	0-20 (nominal)	5.0	1.0	0.55	0.20
Naphthalene	8260B	51-131	62-126	0-30 (nominal)	0-20 (nominal)	5.0	1.0	0.88	0.30
1,2,3-Trichlorobenzene	8260B	55-134	70-122	0-30 (nominal)	0-20 (nominal)	5.0	1.0	0.76	0.27
Methyl Acetate	8260B	72-133	70-132	0-30 (nominal)	0-20 (nominal)	5.0	1.0	2.7	0.53
Methylcyclohexane	8260B	71-127	73-125	0-30 (nominal)	0-20 (nominal)	5.0	1.0	0.96	0.30
Alkylbenzenes (Total)	8260B	79-118	85-119	0-30 (nominal)	0-20 (nominal)	35	7.0	0.67	0.19
Surrogates:									

Target List	EPA Method	Soil Accuracy % Recovery	Aqueous Accuracy % Recovery	Soil Precision RPD (1)	Aqueous Precision RPD (1)	Soil PQLs	Water PQLs	Soil MDLs	Water MDLs
DBFM	8260B	64-130	68-128	--	--	--	--	--	--
1,2-DCA-d4	8260B	58-134	67-135	--	--	--	--	--	--
Toluene-d8	8260B	67-118	65-128	--	--	--	--	--	--
BFB	8260B	47-119	56-133	--	--	--	--	--	--
Semivolatile Organic Compounds						**ug/Kg**	**ug/L**	**ug/Kg**	**ug/L**
1,4-Dioxane	8270C	15-59	10-73	0-50 (nominal)	0-30 (nominal)	330	10	54	1.8
n-Nitrosodimethylamine	8270C	43-115	11-95	0-50 (nominal)	0-30 (nominal)	330	10	87	2.2
Pyridine	8270C	22-64	10-96	0-50 (nominal)	0-30 (nominal)	1600	50	106	1.5
2-Picoline	8270C	30-150 (nominal)	30-150 (nominal)	0-50 (nominal)	0-30 (nominal)	330	10	113	3.1
n-Nitrosomethylethylamine	8270C	30-150 (nominal)	30-150 (nominal)	0-50 (nominal)	0-30 (nominal)	330	10	151	1.5
Methyl Methanesulfonate	8270C	30-150 (nominal)	30-150 (nominal)	0-50 (nominal)	0-30 (nominal)	330	10	194	3.5
n-Nitrosodiethylamine	8270C	30-150 (nominal)	30-150 (nominal)	0-50 (nominal)	0-30 (nominal)	330	10	151	1.1
Ethyl Methanesulfonate	8270C	30-150 (nominal)	30-150 (nominal)	0-50 (nominal)	0-30 (nominal)	330	10	199	3.6
Benzaldehyde	8270C	10-123	10-189	0-50 (nominal)	0-30 (nominal)	330	10	120	1.0
Aniline	8270C	25-104	10-59	0-50 (nominal)	0-30 (nominal)	820	25	91	0.6
Phenol	8270C	56-108	10-78	0-50 (nominal)	0-30 (nominal)	330	10	156	1.8
Bis (2-Chloroethyl) Ether	8270C	50-104	45-95	0-50 (nominal)	0-30 (nominal)	330	10	81	2.0
2-Chlorophenol	8270C	57-102	44-91	0-50 (nominal)	0-30 (nominal)	330	10	164	3.2
1,3-Dichlorobenzene	8270C	58-91	37-90	0-50 (nominal)	0-30 (nominal)	330	10	78	2.1
1,4-Dichlorobenzene	8270C	61-94	38-91	0-50 (nominal)	0-30 (nominal)	330	10	86	2.2
1,2-Dichlorobenzene	8270C	65-93	39-94	0-50 (nominal)	0-30 (nominal)	330	10	88	2.4
Benzyl Alcohol	8270C	63-109	16-118	0-50 (nominal)	0-30 (nominal)	660	20	57	1.2
2,2'-Oxybis(1-chloropropane	8270C	61-98	42-100	0-50 (nominal)	0-30 (nominal)	330	10	89	2.1
2-Methylphenol	8270C	60-99	37-87	0-50 (nominal)	0-30 (nominal)	330	10	200	3.8
Acetophenone	8270C	59-102	49-102	0-50 (nominal)	0-30 (nominal)	330	10	178	3.9
n-Nitrosopyrrolidine	8270C	30-150 (nominal)	30-150 (nominal)	0-50 (nominal)	0-30 (nominal)	330	10	167	1.3
o-Toluidine	8270C	30-150 (nominal)	30-150 (nominal)	0-50 (nominal)	0-30 (nominal)	820	25	143	1.1
n-Nitroso-di-n-propylamine	8270C	52-93	41-97	0-50 (nominal)	0-30 (nominal)	330	10	83	1.9
n-Nitrosomorpholine	8270C	30-150 (nominal)	30-150 (nominal)	0-50 (nominal)	0-30 (nominal)	330	10	152	1.1
3&4-Methylphenol	8270C	58-105	28-85	0-50 (nominal)	0-30 (nominal)	330	10	187	5.6
Hexachloroethane	8270C	59-90	31-90	0-50 (nominal)	0-30 (nominal)	330	10	96	2.3
Nitrobenzene	8270C	59-103	48-95	0-50 (nominal)	0-30 (nominal)	330	10	91	3.1
n-Nitrosopiperidine	8270C	30-150 (nominal)	30-150 (nominal)	0-50 (nominal)	0-30 (nominal)	330	10	153	1.2
Isophorone	8270C	23-110	53-93	0-50 (nominal)	0-30 (nominal)	330	10	75	1.7
2-Nitrophenol	8270C	61-100	48-101	0-50 (nominal)	0-30 (nominal)	330	10	167	2.7
2,4-Dimethylphenol	8270C	52-99	51-87	0-50 (nominal)	0-30 (nominal)	330	10	165	4.4
O,O,O-Triethylphosphorothioate	8270C	30-150 (nominal)	30-150 (nominal)	0-50 (nominal)	0-30 (nominal)	330	10	159	1.4

Target List	EPA Method	Soil Accuracy % Recovery	Aqueous Accuracy % Recovery	Soil Precision RPD (1)	Aqueous Precision RPD (1)	Soil PQLs	Water PQLs	Soil MDLs	Water MDLs
Bis (2-chloroethoxy)methane	8270C	63-99	40-98	0-50 (nominal)	0-30 (nominal)	330	10	96	2.1
2,4-Dichlorophenol	8270C	63-102	47-106	0-50 (nominal)	0-30 (nominal)	330	10	150	3.0
Benzoic acid	8270C	10-121	10-151	0-50 (nominal)	0-30 (nominal)	820	25	344	3
1,2,4-Trichlorobenzene	8270C	63-93	38-90	0-50 (nominal)	0-30 (nominal)	330	10	81	2.1
Naphthalene	8270C	55-99	48-89	0-50 (nominal)	0-30 (nominal)	330	10	87	2.2
2,6-Dichlorophenol	8270C	65-117	45-109	0-50 (nominal)	0-30 (nominal)	330	10	165	4.0
Hexachloropropene	8270C	30-150 (nominal)	30-150 (nominal)	0-50 (nominal)	0-30 (nominal)	330	10	166	1.3
4-Chloroaniline	8270C	10-89	34-100	0-50 (nominal)	0-30 (nominal)	330	10	119	1.9
Hexachlorobutadiene	8270C	59-90	34-86	0-50 (nominal)	0-30 (nominal)	330	10	83	1.8
n-Nitroso-di-n-butylamine	8270C	30-150 (nominal)	30-150 (nominal)	0-50 (nominal)	0-30 (nominal)	330	10	142	2.0
p-Phenylenediamine	8270C	30-150 (nominal)	30-150 (nominal)	0-50 (nominal)	0-30 (nominal)	330	10	165	10
Caprolactam	8270C	30-140	10-86	0-50 (nominal)	0-30 (nominal)	330	10	144	0.4
4-Chloro-3-methylphenol	8270C	62-109	63-101	0-50 (nominal)	0-30 (nominal)	330	10	166	3.6
a,a-Dimethylphenethylamine	8270C	30-150 (nominal)	30-150 (nominal)	0-50 (nominal)	0-30 (nominal)	820	25	151	16
Safrole	8270C	30-150 (nominal)	30-150 (nominal)	0-50 (nominal)	0-30 (nominal)	330	10	147	2.5
2-Methylnaphthalene	8270C	42-127	50-103	0-50 (nominal)	0-30 (nominal)	330	10	92	3.2
1-Methylnaphthalene		57-93	27-120	0-50 (nominal)	0-30 (nominal)	330	10	124	2.9
Hexachlorocyclopentadiene	8270C	23-107	23-70	0-50 (nominal)	0-30 (nominal)	330	10	82	1.2
1,2,4,5-Tetrachlorobenzene	8270C	30-150 (nominal)	30-150 (nominal)	0-50 (nominal)	0-30 (nominal)	330	10	135	1.8
2,4,6-Trichlorophenol	8270C	61-105	57-109	0-50 (nominal)	0-30 (nominal)	330	10	155	2.7
2,4,5-trichlorophenol	8270C	64-107	53-136	0-50 (nominal)	0-30 (nominal)	820	25	155	3.6
Diethyl adipate	8270C	34-133	10-113	0-50 (nominal)	0-30 (nominal)	330	10	84	1.7
Isosafrole	8270C	30-150 (nominal)	30-150 (nominal)	0-50 (nominal)	0-30 (nominal)	330	10	133	1.5
2-Chloronaphthalene	8270C	68-102	37-76	0-50 (nominal)	0-30 (nominal)	330	10	87	2.9
1,1'-Biphenyl	8270C	50-113	51-105	0-50 (nominal)	0-30 (nominal)	330	10	73	2.7
1-Chloronaphthalene	8270C	30-150 (nominal)	30-150 (nominal)	0-50 (nominal)	0-30 (nominal)	330	10	128	3.9
2-Nitroaniline	8270C	58-120	56-108	0-50 (nominal)	0-30 (nominal)	820	25	75	1.8
1,4-Naphthoquinone	8270C	30-150 (nominal)	30-150 (nominal)	0-50 (nominal)	0-30 (nominal)	330	10	169	8.2
Dimethyl phthalate	8270C	66-121	10-111	0-50 (nominal)	0-30 (nominal)	330	10	78	2.01
1,3-Dinitrobenzene	8270C	30-150 (nominal)	30-150 (nominal)	0-50 (nominal)	0-30 (nominal)	330	10	142	1.4
2,6-Dinitrotoluene	8270C	64-109	65-110	0-50 (nominal)	0-30 (nominal)	330	10	79	2.0
Acenaphthylene	8270C	59-108	59-97	0-50 (nominal)	0-30 (nominal)	330	10	70	1.5
3-Nitroaniline	8270C	10-173	46-97	0-50 (nominal)	0-30 (nominal)	820	25	94	1.5
Acenaphthene	8270C	64-106	58-99	0-50 (nominal)	0-30 (nominal)	330	10	65	1.5
2,4-Dinitrophenol	8270C	20-81	12-143	0-50 (nominal)	0-30 (nominal)	820	25	377	1
Pentachlorobenzene	8270C	30-150 (nominal)	30-150 (nominal)	0-50 (nominal)	0-30 (nominal)	330	10	164	1.5
Dibenzofuran	8270C	60-108	62-104	0-50 (nominal)	0-30 (nominal)	330	10	79	1.6

Target List	EPA Method	Soil Accuracy % Recovery	Aqueous Accuracy % Recovery	Soil Precision RPD (1)	Aqueous Precision RPD (1)	Soil PQLs	Water PQLs	Soil MDLs	Water MDLs
4-Nitrophenol	8270C	45-139	10-114	0-50 (nominal)	0-30 (nominal)	820	25	309	1.8
2,4-Dinitrotoluene	8270C	64-111	66-123	0-50 (nominal)	0-30 (nominal)	330	10	85	2.2
1-Naphthylamine	8270C	30-150 (nominal)	30-150 (nominal)	0-50 (nominal)	0-30 (nominal)	330	10	71	1.2
2,3,4,6-Tetrachlorophenol	8270C	58-93	49-119	0-50 (nominal)	0-30 (nominal)	330	10	140	2.7
2-Naphthylamine	8270C	30-150 (nominal)	30-150 (nominal)	0-50 (nominal)	0-30 (nominal)	330	10	163	1.2
Diethylphthalate	8270C	59-113	58-101	0-50 (nominal)	0-30 (nominal)	330	10	80	2.0
Fluorene	8270C	57-108	63-107	0-50 (nominal)	0-30 (nominal)	330	10	81	2.1
4-Chlorophenyl-phenylether	8270C	67-110	65-100	0-50 (nominal)	0-30 (nominal)	330	10	78	2.2
O,O-Diethyl-o-2-pyrazinylphosphoroth	8270C	30-150 (nominal)	30-150 (nominal)	0-50 (nominal)	0-30 (nominal)	660	20	185	1.3
5-Nitro-O-toluidine	8270C	30-150 (nominal)	30-150 (nominal)	0-50 (nominal)	0-30 (nominal)	330	10	177	3.4
4-Nitroaniline	8270C	40-142	52-106	0-50 (nominal)	0-30 (nominal)	820	25	134	1.6
4,6-Dinitro-2-methylphenol	8270C	40-111	52-129	0-50 (nominal)	0-30 (nominal)	820	25	337	2.0
n-Nitrosodiphenylamine	8270C	12-145	52-96	0-50 (nominal)	0-30 (nominal)	330	10	219	3.7
1,2-Diphenylhydrazine	8270C	53-119	57-98	0-50 (nominal)	0-30 (nominal)	660	20	142	2.0
Azobenzene	8270C	58-121	57-98	0-50 (nominal)	0-30 (nominal)	660	20	138	1.9
Sulfotepp	8270C	30-150 (nominal)	30-150 (nominal)	0-50 (nominal)	0-30 (nominal)	330	10	185	1.6
Diallate	8270C	30-150 (nominal)	30-150 (nominal)	0-50 (nominal)	0-30 (nominal)	330	10	181	1.6
Phorate	8270C	30-150 (nominal)	30-150 (nominal)	0-50 (nominal)	0-30 (nominal)	330	10	140	0.9
1,3,5-Trinitrobenzene	8270C	30-150 (nominal)	30-150 (nominal)	0-50 (nominal)	0-30 (nominal)	820	10	344	3.1
4-Bromophenyl-phenylether	8270C	67-119	56-106	0-50 (nominal)	0-30 (nominal)	330	10	85	1.9
Phenacetin	8270C	30-150 (nominal)	30-150 (nominal)	0-50 (nominal)	0-30 (nominal)	330	10	187	1.4
Hexachlorobenzene	8270C	65-112	51-112	0-50 (nominal)	0-30 (nominal)	330	10	82	2.1
Dimethoate	8270C	30-150 (nominal)	30-150 (nominal)	0-50 (nominal)	0-30 (nominal)	330	10	210	0.6
Atrazine	8270C	55-135	83-153	0-50 (nominal)	0-30 (nominal)	330	10	91	3.3
Pentachlorophenol	8270C	57-125	41-134	0-50 (nominal)	0-30 (nominal)	820	25	237	2.3
Pentachloronitrobenzene	8270C	30-150 (nominal)	30-150 (nominal)	0-50 (nominal)	0-30 (nominal)	330	10	179	1.6
4-Aminobiphenyl	8270C	30-150 (nominal)	30-150 (nominal)	0-50 (nominal)	0-30 (nominal)	330	10	112	0.8
Proamide	8270C	30-150 (nominal)	30-150 (nominal)	0-50 (nominal)	0-30 (nominal)	330	10	177	1.9
Phenanthrene	8270C	61-117	70-107	0-50 (nominal)	0-30 (nominal)	330	10	83	2.4
Dinoseb	8270C	30-150 (nominal)	30-150 (nominal)	0-50 (nominal)	0-30 (nominal)	330	10	208	1.5
Disulfoton	8270C	30-150 (nominal)	30-150 (nominal)	0-50 (nominal)	0-30 (nominal)	330	10	180	1.9
Anthracene	8270C	56-114	67-112	0-50 (nominal)	0-30 (nominal)	330	10	84	1.7
Carbazole	8270C	49-168	57-125	0-50 (nominal)	0-30 (nominal)	330	10	111	2.1
Methyl parathion	8270C	30-150 (nominal)	30-150 (nominal)	0-50 (nominal)	0-30 (nominal)	330	10	209	1.1
Di-n-butylphthalate	8270C	56-129	68-114	0-50 (nominal)	0-30 (nominal)	330	10	101	2.5
4-Nitroquinoline-1-oxide	8270C	30-150 (nominal)	30-150 (nominal)	0-50 (nominal)	0-30 (nominal)	820	25	255	8.9
Parathion	8270C	30-150 (nominal)	30-150 (nominal)	0-50 (nominal)	0-30 (nominal)	820	25	193	1.4

Target List	EPA Method	Soil Accuracy % Recovery	Aqueous Accuracy % Recovery	Soil Precision RPD (1)	Aqueous Precision RPD (1)	Soil PQLs	Water PQLs	Soil MDLs	Water MDLs
Methapyrilene	8270C	30-150 (nominal)	30-150 (nominal)	0-50 (nominal)	0-30 (nominal)	330	25	118	12.3
Isodrin	8270C	30-150 (nominal)	30-150 (nominal)	0-50 (nominal)	0-30 (nominal)	330	10	172	1.6
Fluoranthene	8270C	70-115	67-119	0-50 (nominal)	0-30 (nominal)	330	10	106	2.4
Benzidine	8270C	10-150	10-100 (nominal)	0-50 (nominal)	0-30 (nominal)	1500	50	100	16.2
Pyrene	8270C	54-131	58-135	0-50 (nominal)	0-30 (nominal)	330	10	101	1.9
Aramite	8270C	30-150 (nominal)	30-150 (nominal)	0-50 (nominal)	0-30 (nominal)	660	20	156	4.3
p-Dimethylaminoazobenzene	8270C	30-150 (nominal)	30-150 (nominal)	0-50 (nominal)	0-30 (nominal)	330	10	210	1.1
Chlorobenzilate	8270C	30-150 (nominal)	30-150 (nominal)	0-50 (nominal)	0-30 (nominal)	330	10	243	1.0
Famphur	8270C	30-150 (nominal)	30-150 (nominal)	0-50 (nominal)	0-30 (nominal)	990	30	680	22.3
Kepone	8270C	30-150 (nominal)	30-150 (nominal)	0-50 (nominal)	0-30 (nominal)	820	25	165	13
3,3'-Dimethylbenzidine	8270C	30-150 (nominal)	30-150 (nominal)	0-50 (nominal)	0-30 (nominal)	330	25	114	12
Butylbenzylphthalate	8270C	50-131	56-129	0-50 (nominal)	0-30 (nominal)	330	10	93	1.9
Bis(2-ethylhexyl)adipate	8270C	39-130	50-142	0-50 (nominal)	0-30 (nominal)	330	10	98	1.7
2-Acetylaminofluorene	8270C	30-150 (nominal)	30-150 (nominal)	0-50 (nominal)	0-30 (nominal)	330	10	188	1.4
Benzo(a)anthracene	8270C	65-111	66-109	0-50 (nominal)	0-30 (nominal)	330	10	86	1.6
3,3'-Dichlorobenzidine	8270C	10-130	36-87	0-50 (nominal)	0-30 (nominal)	330	10	114	1.1
Chrysene	8270C	66-107	68-114	0-50 (nominal)	0-30 (nominal)	330	10	95	1.7
Bis(2-ethylhexyl)phthalate	8270C	42-142	51-155	0-50 (nominal)	0-30 (nominal)	330	10	98	1.8
Di-n-octylphthalate	8270C	10-187	33-184	0-50 (nominal)	0-30 (nominal)	330	10	211	1.8
Benzo(b)fluoranthene	8270C	47-121	60-108	0-50 (nominal)	0-30 (nominal)	330	10	134	1.2
7,12-Dimethylbenz(a)anthracene	8270C	30-150 (nominal)	30-150 (nominal)	0-50 (nominal)	0-30 (nominal)	330	10	178	1.8
Benzo(k)fluoranthene	8270C	37-130	59-125	0-50 (nominal)	0-30 (nominal)	330	10	83	1.6
Benzo(a)pyrene	8270C	62-114	63-118	0-50 (nominal)	0-30 (nominal)	330	10	93	1.2
Hexachlorophene	8270C	30-150 (nominal)	30-150 (nominal)	0-50 (nominal)	0-30 (nominal)	330	25	165	5.0
3-Methylcholanthrene	8270C	30-150 (nominal)	30-150 (nominal)	0-50 (nominal)	0-30 (nominal)	330	10	227	1.9
Dibenz(a,j)acridine	8270C	30-150 (nominal)	30-150 (nominal)	0-50 (nominal)	0-30 (nominal)	330	10	166	1.7
Indeno(1,2,3-cd)pyrene	8270C	55-128	55-111	0-50 (nominal)	0-30 (nominal)	330	10	122	1.9
Dibenzo(a,h)anthracene	8270C	54-133	61-111	0-50 (nominal)	0-30 (nominal)	330	10	128	1.7
Benzo(g,h,i)perylene	8270C	53-126	58-115	0-50 (nominal)	0-30 (nominal)	330	10	104	1.5
Surrogates:									
2-Fluorophenol	8270C	43-99	10-80	---	---	---	---	---	---
Phenol-d6	8270C	53-98	10-90	---	---	---	---	---	---
Nitrobenzene-d5	8270C	47-100	41-91	---	---	---	---	---	---
2-Fluorobiphenyl	8270C	49-114	43-90	---	---	---	---	---	---
2,4,6-Tribromophenol	8270C	44-111	37-112	---	---	---	---	---	---
Terphenyl-d14	8270C	58-140	36-156	---	---	---	---	---	---
Metals -ICP						**mg/Kg**	**ug/L**	**mg/Kg**	**ug/L**

Target List	EPA Method	Soil Accuracy % Recovery	Aqueous Accuracy % Recovery	Soil Precision RPD (1)	Aqueous Precision RPD (1)	Soil PQLs	Water PQLs	Soil MDLs	Water MDLs
Aluminum	6010	Manufacturer (6)	80-120	0-20 (nominal)	0-20 (nominal)	30	300	1.6	15
Antimony	6010	Manufacturer (6)	80-120	0-20 (nominal)	0-20 (nominal)	0.80	8.0	0.110	1.5
Arsenic	6010	Manufacturer (6)	80-120	0-20 (nominal)	0-20 (nominal)	0.80	8.0	0.110	1.9
Barium	6010	Manufacturer (6)	80-120	0-20 (nominal)	0-20 (nominal)	0.50	5.0	0.026	0.44
Beryllium	6010	Manufacturer (6)	80-120	0-20 (nominal)	0-20 (nominal)	0.50	5.0	0.0085	0.042
Boron	6010	Manufacturer (6)	80-120	0-20 (nominal)	0-20 (nominal)	10	100	0.08	1.7
Cadmium	6010	Manufacturer (6)	80-120	0-20 (nominal)	0-20 (nominal)	1.0	10	0.0084	0.04
Calcium	6010	Manufacturer (6)	80-120	0-20 (nominal)	0-20 (nominal)	10	100	1.1	5.8
Chromium	6010	Manufacturer (6)	80-120	0-20 (nominal)	0-20 (nominal)	1.5	15	0.032	0.32
Cobalt	6010	Manufacturer (6)	80-120	0-20 (nominal)	0-20 (nominal)	3.0	30	0.019	0.28
Copper	6010	Manufacturer (6)	80-120	0-20 (nominal)	0-20 (nominal)	2.5	25	0.073	0.48
Iron	6010	Manufacturer (6)	80-120	0-20 (nominal)	0-20 (nominal)	10	100	0.3	6.3
Lead	6010	Manufacturer (6)	80-120	0-20 (nominal)	0-20 (nominal)	0.5	5.0	0.10	0.73
Magnesium	6010	Manufacturer (6)	80-120	0-20 (nominal)	0-20 (nominal)	10	100	0.36	4.8
Manganese	6010	Manufacturer (6)	80-120	0-20 (nominal)	0-20 (nominal)	0.50	5.0	0.058	0.37
Mercury	7471/7470	Manufacturer (6)	80-120	0-20 (nominal)	0-20 (nominal)	0.040	0.20	0.0011	0.037
Molybdenum	6010	Manufacturer (6)	80-120	0-20 (nominal)	0-20 (nominal)	1.0	10	0.050	0.92
Nickel	6010	Manufacturer (6)	80-120	0-20 (nominal)	0-20 (nominal)	4.0	40	0.054	0.29
Potassium	6010	Manufacturer (6)	80-120	0-20 (nominal)	0-20 (nominal)	100	1000	10.0	105
Selenium	6010	Manufacturer (6)	80-120	0-20 (nominal)	0-20 (nominal)	1.0	10	0.39	3.7
Silicon	6010	Manufacturer (6)	80-120	0-20 (nominal)	0-20 (nominal)	20	200	1.2	21
Silver	6010	Manufacturer (6)	80-120	0-20 (nominal)	0-20 (nominal)	1.5	15	0.049	0.48
Sodium	6010	Manufacturer (6)	80-120	0-20 (nominal)	0-20 (nominal)	100	1000	1.7	34
Strontium	6010	Manufacturer (6)	80-120	0-20 (nominal)	0-20 (nominal)	10	100	0.016	0.15
Thallium	6010	Manufacturer (6)	80-120	0-20 (nominal)	0-20 (nominal)	1.5	15	0.160	0.67
Tin	6010	Manufacturer (6)	80-120	0-20 (nominal)	0-20 (nominal)	10	100	0.16	1.1
Vanadium	6010	Manufacturer (6)	80-120	0-20 (nominal)	0-20 (nominal)	2.5	25	0.035	0.39
Zinc	6010	Manufacturer (6)	80-120	0-20 (nominal)	0-20 (nominal)	2.5	25	0.063	0.51

LCS and Surrogate limits are updated annually

MDL's are updated annually

1. Represented as Relative Percent Difference
2. Accuracy is determined by Laboratory Control Samples and Matrix Spike/Matrix Spike Duplicates.
3. Accuracy for metals for MS/MSD only applies when the spike is > 4X the native analyte concentration.
4. Practical Quantitation Limits (PQLs) can increase based on percent water content and/or dilution factors.
5. The MDL and PQL for method 6020 include a dilution factor of 5 due to the matrix interference of Hydrochloric acid.
6. LCS limits are based on the manufacturer's limits for the SRM sample. Limits for MS/MSD are nominal (75-125%)

Target List	EPA Method	Soil Accuracy % Recovery	Aqueous Accuracy % Recovery	Soil Precision RPD (1)	Aqueous Precision RPD (1)	Soil PQLs	Water PQLs	Soil MDLs	Water MDLs

7. MDL's are determined annually therefore the MDLs listed on this table may change.
8. DoD QSM Version 4.1 limits

KATAHDIN ANALYTICAL SERVICES, INC.
STANDARD OPERATING PROCEDURE

SOP Number: CA-202
Revision History
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Page 1

TITLE: ANALYSIS OF VOAs BY PURGE AND TRAP GC/MS: SW-846 METHOD 8260

Prepared By: GC/MS Group Date: 2/97

Approved By:

Group Supervisor: JA Halay Date: 011201

Operations Manager: for C. Burton Date: 1/15/01

QA Officer: Deborah J. Nadeau Date: 1·23·01

General Manager: Deborah F. Nadeau Date: 1/16/01

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
03 8260B	Format changes, added pollution prevention, changes to calibration section, new limits, added instrument. Other minor changes throughout.	DN	1·23·01	1·23·01
04 8260B	Revised sections 7.5.3.1, 7.5.5, 7.7.1, 7.8.2 + Table 2 to comply with South Carolina. Added NH oxygenates to calibration.	DN	5·23·01	5·23·01
05 8260B	Updated VOA calibration standard mixes. Added statistical limits for LCS/MS/MSD recoveries and the updated corrective actions	DN	5·21·02	5·21·02
06 8260B	Reorganization of sections 4, 5, 6 and 7, and Tables and Figures. Added definitions and information for the new data processing system.	MRC	05.03.04	05.03.04
07 8260B	minor changes rewording of sec 1.7.6.3 preservation of calcareous soils	LAD	020305	020305

TITLE: ANALYSIS OF VOAs BY PURGE AND TRAP GC/MS: SW-846 METHOD 8260

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
08 8260B	Added references, setup and operation for the Encon / Centurion autosampler / Purge and Trap. Added ref. to instrument "T" and removed instrument "Q". Edited Std. conc. to reflect new instrumentation. Minor changes throughout to reflect current practice and correct typos.	LAD	04/06	04/06
09 8260B	Sect. 4H - added list of waste streams generated and location of satellites. Clarified RT window studies. Added reference to MI Sop. Removed Grand mean Calibration model. Added wording for project specific acceptance criteria. Added LCS marginal outlier criteria. Added wording clarifying calibration verification Std. Criteria and corrective action. Reworded Correlation	LAD	LAD 7-25-07 ~~03/07~~ 07/07	~~03/07~~ 07/07
10	Coefficient criteria Updated Sections 7.4.5, 7.4.6, 7.4.7, 7.5.2, 8.1, 10.0 and Table 1 with DoD QSM version 4.1 criteria	LAD	08/09	08/09
11	Added Table 2 with DoD QSM V. 4.1 QC Requirements. Added if the MSD Batch requirement cannot be fulfilled, a LCSD must be analyzed. Removed "2" instrument and added the "C" and "D" instruments.	LAD	04/10	04/10

TITLE: **ANALYSIS OF VOAs BY PURGE AND TRAP GC/MS: SW-846 METHOD 8260**

Please acknowledge receipt of this standard operating procedure by signing and dating both of the spaces provided. Return the bottom half of this sheet to the QA Department.

I acknowledge receipt of copy ___ of document **SOP CA-202-11**, titled **ANALYSIS OF VOAs BY PURGE AND TRAP GC/MS: SW-846 METHOD 8260**.

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STANDARD OPERATING PROCEDURE

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TITLE: **ANALYSIS OF VOAs BY PURGE AND TRAP GC/MS: SW-846 METHOD 8260**

1.0 SCOPE AND APPLICATION

The purpose of this SOP is to describe the procedures utilized by Katahdin Analytical Services, Inc. laboratory personnel to prepare and analyze aqueous and solid matrix samples for purgeable organics by GC/MS in accordance with SW-846 Method 8260, current revision.

This SOP will consolidate all aspects of the analyses in one working document, to be revised as necessary, for the purposes of consistency in data quality.

1.1 Definitions

VOC: Volatile Organic Compounds

VOA: Volatile Organic Analysis

ANALYTICAL BATCH: 20 or fewer samples that are analyzed together with the same method sequence and the same lots of reagents and with the handling practices common to each sample within the same time period or in continuous sequential time periods.

METHOD BLANK (laboratory reagent blank): A quality control sample designed to determine if method analytes or other interferences are present in the laboratory environment, the reagents, or the apparatus. Laboratory reagent grade water is used as a blank matrix. The blank is taken through the appropriate steps of the process.

CALIBRATION CHECK: Verification of the ratio of instrument response to analyte amount, a calibration check is done by analyzing a mid point standard. The calibration check verifies that instrument conditions are sufficiently similar to those at initial calibration.

CALIBRATION STANDARD (WORKING STANDARD): A solution prepared from the stock standard solution that is used to calibrate the instrument response with respect to analyte concentration.

INDEPENDANT CALIBRATION STANDARD: A solution prepared from a stock standard solution independent of the standard that is used to calibrate the instrument. This is prepared as an LCS and analyzed after the calibration before any sample analysis.

LABORATORY CONTROL SAMPLE (LCS): A blank that has been spiked with the analyte(s) from an independent source and is analyzed exactly like a sample. Its purpose is to determine whether the methodology is in control and to measure the degree of accuracy of the determination.

MATRIX SPIKE/MATRIX SPIKE DUPLICATE (MS/MSD): Predetermined quantities of stock solutions containing target analytes are added to a sample matrix prior to sample

extraction, in the case of soils, and/or analysis. Samples are split into duplicates, spiked and analyzed. Percent recoveries are calculated for each of the spiked analytes. The relative percent difference between the samples is calculated and used to assess analytical precision.

STANDARD CURVE (CALIBRATION CURVE): A curve that plots concentration of known analyte standard versus the instrument response to the analyte.

STOCK STANDARD SOLUTION: A concentrated solution containing a single analyte or mix of certified standards, or a concentrated solution of a single analyte prepared in the laboratory with an assay reference compound. Stock standard solutions are used to prepare calibration standards.

SURROGATES: Organic compounds which are similar to analytes of interest in chemical composition as well as extraction and chromatography characteristics, but which are not normally found in environmental samples. These compounds are spiked into all blanks, standards, samples and spiked samples prior to analysis. Percent recoveries are calculated for each surrogate. Surrogates provide an indication of the accuracy for the analytical determination in a discrete sample matrix.

TARGET: A software system that combines full processing, reporting and comprehensive review capabilities, regardless of chromatographic vendor and data type.

TARGET DB: An oracle database used to store and organize all Target data files.

QUICKFORMS: A laboratory reporting software for Target and Target DB. The QuickForms report module for Target is preconfigured with generalized forms and US EPA CLP report forms and disk deliverables, which can be customized.

1.2 Responsibilities

This method is restricted to use by, or under the supervision of analysts experienced in the analysis of volatile organics by the current revision of EPA Method 8260. Each analyst must demonstrate and document their ability to generate acceptable results with this method. Refer to Katahdin SOP QA-805, current revision, “Personnel Training and Demonstration of Capability”.

It is the responsibility of all Katahdin technical personnel involved in analysis of volatile organics by Method 8260 to read and understand this SOP, to adhere to the procedures outlined, and to properly document their data in the appropriate logbook. Any deviations from the test or irregularities with the samples should also be recorded in the lab logbook and reported to the Department Manager or designated qualified data reviewer responsible for this data.

It is the responsibility of the Department Manager to oversee that members of their group follow this SOP, to ensure that their work is properly documented and to initiate periodic review of the associated logbooks.

1.3 Safety

Users of this procedure must be cognizant of inherent laboratory hazards, proper disposal procedures for contaminated materials and appropriate segregation of hazardous wastes. The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical should be treated as a potential health hazard. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Everyone involved with the procedure must be familiar with the MSDSs (material safety data sheets) for all the materials used in this procedure.

Each qualified analyst or technician must be familiar with Katahdin Analytical Health and Safety Manual including the Katahdin Hazardous Waste Management Plan and must follow appropriate procedures. These include the use of appropriate personal protective equipment (PPE) such as safety glasses, gloves and lab coats when working with chemicals or near an instrument and not taking food or drink into the laboratory. Each analyst should know the location of all safety equipment. Each analyst shall receive a safety orientation from their Department Manager, or designee, appropriate for the job functions they will perform.

1.4 Pollution Prevention/Waste Disposal

Whenever possible, laboratory personnel should use pollution prevention techniques to address their waste generation. Refer to the current revision of the Katahdin Hazardous Waste Management Program for further details on pollution prevention techniques.

After analysis, partially-filled VOA vials and sample jars are returned to the appropriate refrigerators to be disposed of in adherence with the Katahdin Hazardous Waste Management Plan and Safety Manual and SOP SD-903, Sample Disposal, current revision. Expired standards are lab packed, placed in the Katahdin hazardous waste storage area, and disposed of in accordance with this SOP SD-903.

Sample aliquots used for analysis are disposed of in accordance with SOP SD-903 and the Katahdin Hazardous Waste Management Plan and Safety Manual. The soil samples must be decanted and the soil fraction disposed of separately in compliance with Katahdin's disposal policies.
There are three general types of waste generated while performing the 8260 method. The "K" waste is a combination of water, sample aliquot (post analysis), as well as internal and surrogate standards. "K" waste is generated when preparing QC, during sample analysis, and procedural cleanup. There are "K" satellites

attached to each GC/MS instrument as well as an additional satellite located adjacent to the VOA sample preparation bench. "O" waste consists of methanol (as well as trace amounts of volatile analytes) and is generated when standard preparation syringes are rinsed three times with methanol. The "O" waste stream satellite is located inside the fume hood. Organic soil waste stream "I" consists of any solid left over from sample preparation and/or analysis and is located inside the fume hood. All satellites listed above are stored in a secondary container and are located in the Volatile Organics Laboratory room 111.

2.0 SUMMARY OF METHOD

The general methodology involves purging aqueous and soil samples with helium, an inert gas, for a set period of time to efficiently transfer purgeable organics to the gaseous phase. Soil samples with higher contaminant levels are extracted with methanol prior to the helium purge. These volatile organics are then retained on a cooled trap (commercially available trap suitable for the methodology) before heating causes desorption into a gas chromatograph for compound separation. Detection occurs with an electron impact ionization mass spectrometer.

3.0 INTERFERENCES

Interfering contamination may occur when a sample containing low concentrations of VOCs is analyzed immediately after a sample containing high concentrations of VOCs. During initial data review, all analyses are evaluated for potential carryover. Any samples that have suspected carryover are reanalyzed. GC/MS policy is to reanalyze a sample with positive detects greater than the Practical Quantitation Limit (PQL) that has been run immediately after a sample with the same positive detects over the upper limit of the calibration. Typically 2 or 3 rinsing blanks are analyzed at the end of a sequence. Samples are not analyzed on the instrument until a blank with no detects above PQL can be obtained. If the lines are determined to be contaminated, then the entire Tekmar or Archon must be backflushed with warm methanol and water.

4.0 APPARATUS AND MATERIALS

4.1 GC: Hewlett Packard 6890 & 5890

4.2 Mass Spectrometers (MS): HP5973, HP5972 and HP5970

4.3 Helium: Carrier gas for routine applications. All carrier gas lines must be constructed from stainless steel or copper tubing; non-polytetrafluoroethylene (non-PTFE) thread sealant or flow controllers with rubber component are not to be used.

4.4 Columns: RTX-VMS, 40 meter, 0.18 mm ID or equivalent.

4.5 Purge and Traps: Archon 5100, Tekmar 2016 and Centurion auto samplers, and Tekmar 2000, 3000 and Encon concentrators.

4.6 Purge tubes: 5 mL fritted and 25 mL fritted purge vessels and 40 mL VOA vials for soil analysis.

4.7 Hamilton Gastight syringes: 2.00 uL to 25.00 mL.

4.8 Acquisition System: The acquisition system must be interfaced to the MS and allow continuous acquisition of data throughout the duration of the chromatographic program. It must permit, at a minimum, the output of time vs. intensity (peak height or peak area). Hewlett Packard Chemstation or equivalent.

4.9 Data System: The Target software is used for processing data and generating forms.

5.0 REAGENTS

5.1 Purge and trap grade methanol

5.2 Organic-free Laboratory reagent grade water: Siemens, Poland Spring, or equivalent. This water may need to be purged with nitrogen to eliminate organic contaminants such as Methylene chloride and Chloroform, which are commonly found at ambient levels in the laboratory.

5.3 Standards: Stock standards and working standards are received and recorded in accordance with SOP CA-106 “Standard Preparation and Documentation”.

5.3.1 The expiration date for all standards is six months from date of opening the ampule with the following exceptions:

Volatile gases expire within 2 weeks of opening ampule (gases are dichlorodifluoromethane, chloromethane, bromomethane, vinyl chloride, chloroethane, and trichlorofluoromethane).

New standards must be opened if degradation is observed.

5.3.2 Secondary dilution standards

5.3.2.1 Calibration Mix – Prepare a standard in purge and trap methanol containing the compounds listed below. The final concentration of each compound is 200 ug/mL (some individual analyte concentrations may vary, i.e. Ketones). The standard should be prepared in a 1.0 mL conical vial with a mini-inert valve cap. The standard must be prepared every 7 days and stored in the VOA standards freezer between uses.

Acetone	Dibromochloromethane	P-Isopropyltoluene
Benzene	1,2-Dibromoethane	Methylene Chloride
Bromobenzene	Dibromomethane	4-Methyl-2-Pentanone
Bromochloromethane	1,2-Dichlorobenzene	Naphthalene
Bromodichloromethane	1,3-Dichlorobenzene	N-Propylbenzene
Bromoform	1,4-Dichlorobenzene	Styrene
Bromomethane	Dichlorodifluoromethane	1,1,1,2-Tetrachloroethane
2-Butanone	1,1-Dichloroethane	1,1,2,2-Tetrachloroethane
n-Butylbenzene	1,2-Dichloroethane	Tetrachloroethene
sec-Butylbenzene	1,1-Dichloroethene	Tetrahydrofuran
tert-Butylbenzene	cis-1,2-Dichloroethene	Toluene
Carbon Disulfide	Trans-1,2-Dichloroethene	1,2,3-Trichlorobenzene
Carbon Tetrachloride	1,2-Dichloropropane	1,2,4-Trichlorobenzene
Chlorobenzene	1,3-Dichloropropane	1,1,1-Trichloroethane
Chloroethane	2,2-Dichloropropane	1,1,2-Trichloroethane
2-Chloroethylvinyl Ether	1,1-Dichloropropene	Trichloroethene
Chloroform	Cis-1,3-Dichloropropene	Trichlorofluoromethane
Chloromethane	Trans-1,3-Dichloropropene	1,2,3-Trichloropropane
2-Chlorotoluene	Ethylbezene	1,2,4-Trimethylbenzene
4-Chlorotoluene	Hexachlorobutadiene	Vinyl Acetate
Cyclohexane	2-Hexanone	Vinyl Chloride
1,2-Dibromo-3-Chloropropane	Idomethane	1,3,5-Trimethylbenzene
Isopropylbenzene	Methyl Tert-Butyl Ether	1-Chlorohexane

5.3.2.2 Extras mix – Prepare a standard as above containing the compounds listed below. The final concentration of each compound is 200 ug/mL. The standard should be prepared in a 1.0 mL conical vial with a mini-inert valve cap. The standard must be prepared every 30 days and stored in the VOA standards freezer between uses.

Acetonitrile	Isobutyl Alcohol
Acrolein	Methacrylonitrile
Acrylonitrile	Methylcyclohexane
Allyl Chloride	Methyl Acetate
Chloroprene	Methyl Methacrylate
Diethyl Ether	Methyl Tert-Butyl Ether
Cis-1,4-Dichloro-2-Butene	Pentachloroethane
Trans-1,4-Dichloro-2-Butene	Propionitrile
1,4-Dioxane	Tertiary-Amyl Methyl Ether
Di-Isopropyl Ether	Tertiary-Butyl Alcohol
Ethyl Methacrylate	1,3,5-Trichlorobenzene
Ethyl Tertiary-Butyl Ether	1,2,3-Trimethylbenzene
Freon-113	

5.3.2.3 Independent Calibration Verification Standard, Laboratory Control Spike and MS/MSD Mixture - Prepare a standard as above containing the compounds listed in Table 3. The final concentration of each compound is 200 ug/mL (some individual analyte concentrations may vary, i.e. Ketones). The standard should be prepared in a 1.0 mL conical vial with a mini-inert valve cap. The standard must be prepared every 7 days and stored in the VOA standards freezer between uses.

5.3.2.4 Surrogate Spiking Solution - Prepare a standard as above containing the compounds listed below. The final concentration of each compound is 250 ug/mL or 50 ug/mL depending on which autosampler you will be using. The standard must be prepared every 14 days and stored on the Archon and/or the Centurion autosampler in a pressurized vial or in the VOA standards freezer between uses.

4-Bromofluorobenzene
1,2-Dichloroethane-D_4
Toluene-D_8
Dibromofluoromethane

5.3.2.5 Internal Standard Solution - Prepare a standard as above containing the compounds listed below. The final concentration of each compound is 250 ug/mL or 50 ug/mL depending on which autosampler you will be using. The standard must be prepared every 14 days and stored on the Archon and/or the Centurion autosampler in a pressurized vial or in the VOA standards freezer between uses.

Pentafluorobenzene
1,4-Difluorobenzene
Chlorobenzene-D_5
1,4-Dichlorobenzene-D_4

5.3.2.6 BFB Solution - Prepare a standard as above containing 4-BFB. The final concentration is 25 ug/mL. The standard must be prepared every 30 days and stored in the VOA standards freezer between uses.

5.3.2.7 See Table 4 for a complete list of standards, concentration, and vendors.

NOTE: The concentrations of standards may vary depending on the type of autosampler being used.

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

All aqueous samples must be analyzed within 14 days from sample collection if preserved (by addition of HCl to pH <2) or within 7 days from sample collection if unpreserved. All soil/sediments must be analyzed within 14 days from sample collection. For specific projects, soil may be received in pre-weighed vials containing methanol, with an aliquot of the methanol used for analysis. For these projects, the methanol aliquot must be analyzed within 14 days from sample collection. Samples must be stored at $4^{\circ}C \pm 2^{\circ}C$ from the time of receipt at the lab until analysis.

7.0 PROCEDURES

7.1 NAMING AND CODING CONVENTIONS FOR ANALYTICAL STANDARDS – Used in accordance with SOP CA-106 “Standard Preparation and Documentation”.

7.2 COMPUTER (DATA SYSTEM) CONVENTIONS -

Conventions for all instruments are as follows:

Sub-Directory for data acquisition: C:\HPCHEM\1\DATA

Tune file: BFB.U

Method files: I826AXX.M (all samples and standards)
Where:
XX = the calibration number in chronological order
I = instrument ID (C, D, F, M, S or T).
A = matrix (A for water, S for soil and SB for sodium bisulfate soils)

BFB288AQ.M (waters) or BFB288SL.M (soils) (BFB tuning acquisition)

Data files for BFB: IB_ _ _.D where _ _ _ is a number in chronological order from 000 to 999, and I is the instrument ID (C, D, F, M, S or T).

All other data files: I_ _ _ _.D where _ _ _ _ is a number in chronological order from 0000 to 9999, and I is the instrument ID (C, D, F, M, S or T). This file also contains the Quantitation output file.

7.3 INSTRUMENT TUNING - Prior to the analysis of any calibration standards, blanks, or samples, the GC/MS system must be shown to meet the mass spectral ion abundance criteria for a 50 ng injection of p-Bromofluorobenzene (p-BFB), tabulated below:

Mass	Criteria
50	15.0-40.0% of mass 95
75	30.0-60% of mass 95
95	base peak, 100% relative abundance
96	5.0-9.0% of mass 95
173	less than 2.0% of mass 174
174	greater than 50.0% of mass 95
175	5.0-9.0% of mass 174
176	greater than 95.0%, but less than 101.0% of mass 174
177	5.0-9.0% of mass 176

7.3.1 The following are the GC/MS operating conditions for injection of BFB.

GC/MS type: 5970

Column:	RTX-VMS, 40 meter, 0.18 mm ID
Temperatures: Injection port:	170°
Transfer line:	150°
Source:	170°
Analyzer:	170°
Isothermal temperature:	150°
Run time:	10 minutes
Scan start time:	4 minutes
Scan parameters:	not to exceed 2 sec per scan
Mass range:	35-300
Number of A/D samples:	8
GC peak threshold:	1000 counts
Threshold:	10 counts

GC/MS type: 5972 and 5973

Column:	RTX-624, 40 meter, 0.18 mm I.D or RTX-VMS, 40 meter, 0.18 mm ID.
Temperatures: Injection port:	200°
Transfer line:	150°
Detector:	240°
Isothermal temperature:	150°
Run time:	8 minutes
Scan start time:	3 minutes
Scan parameters:	not to exceed 2 sec per scan
Mass range:	35-300
Number of A/D samples:	8
GC peak threshold:	1000 counts
Threshold:	10 counts

The BFB solution must be analyzed once at the beginning of each 12-hour period, the time stamp of the injection of the BFB is the beginning of the 12-hour clock. All calibrations and samples must be run within the 12-hour clock as the method specifies.

When the BFB run has concluded, the run must be evaluated to determine if sample analysis can proceed. The chromatography and the ion ratios must be examined. The BFB run is processed using the current algorithms in the Target software.

If the results indicate the system does not meet acceptance criteria, the GC/MS must be manually tuned. Once the manual tune procedure is completed, BFB must be re-injected and reevaluated. If the instrument still does not meet criteria, notify your Department Manager. Under no circumstances should calibration proceed if the instrument BFB tune is not in criteria.

7.4 INSTRUMENT CONFIGURATION / CALIBRATION

7.4.1 Tekmar LSC 3000/Archon 5100/ Tekmar 2016, Setup/Operation: Please refer to the Tekmar or Archon Manuals for more detailed operations for these instruments.

To begin, set the Tekmar LSC 2000/3000 to the specification listed in section 2-12 of the Archon manual. Edit method 14 as follows:

Method 14 should include:

Standby:	35°
Prepurge:	0 min
Preheat Temp:	0°
Sample Temp:	0°
Purge:	11 min
Dry purge:	2-4 min
Desorb preheat:	245°
Desorb Temp:	250°
Desorb time:	2-5 min
Dry purge:	2-4 min
Bake Time:	10 min
Bake Temp:	260°
Auto drain:	On
Bake gas by pass:	Off
Valve Temp:	120°
Line Temp:	120°
Runs per sample:	1

The above temperature settings are for a Vocarb 3000 trap, these temperatures may vary with the use of alternative traps. Temperature settings may also vary to optimize system performance.

The Archon autosampler should be set up according to the specifications in the manual. The setting of particular concern, with regards to keeping the Tekmar and Archon in coordination with each other, is the desorb time. There are several other programmable features on the Archon; the settings for this feature will depend on the sample matrix and method of analysis. Please refer to the Archon manual for more specifics on its programming features.

7.4.2 Encon/Centurion, Setup/Operation

Please refer to the Encon or Centurion manuals for more detailed operations for the instruments.

To begin, the Encon operation method should contain:

Purge Conditions:
Purge Gas: Helium
Purge Time: 11.0 ±0.1 minute
Purge Flow Rate: approx. 24-40 mL/min
Purge Temperature: Ambient (water)

Desorb Conditions:
DesorbTemp: 250ºC
Desorb Flow rate: 15 mL/min
Desorb Time: 2.0 ± 0.1 min
Bake Time: 10 min
Bake Temperature: 260ºC

The above temperature settings are for a Vocarb 3000 trap, these temperatures may vary with the use of alternative traps. Temperature settings may also vary to optimize system performance.

The Centurion autosampler should be set up according to the specifications in the manual.

7.4.3 Initial Calibration for Method 8260

Once the instrument has achieved BFB tuning criteria, calibration of the instrument can begin.

To determine the linearity of response, the GC/MS must be initially calibrated at six different levels.

For aqueous calibration, target analytes and surrogate are prepared at the following concentrations; 1.0, 5.0, 20, 50, 100 and 200 ug/L. The curve is analyzed at ambient temperature.

For a soil calibration target analytes and surrogates are prepped at the following concentrations: 5.0, 10, 20, 50, 100 and 200 ug/L. The calibration standards are stirred and heated to 40ºC.

The following amounts standards should be added to 100 mL of organic-free laboratory reagent grade water in order to generate a 6-point initial calibration curve:

	STD. ID	CAL. Mix 200 ug/mL	Extras Mix 200 ug/mL	Surr. Mix 250 ug/mL Archon	Surr. Mix 50 ug/mL Centurion
AQ curve only	VSTD001	0.5 uL	0.5 uL	0.4 uL	2.0 uL
	VSTD005	2.5 uL	2.5 uL	2.0 uL	10 uL
SL curve only	VSTD010	5.0 uL	5.0 uL	4.0 uL	20 uL
	VSTD020	10 uL	10 uL	8.0 uL	40 uL
CC	VSTD050	25 uL	25 uL	20 uL	100 uL
	VSTD100	50 uL	50 uL	40 uL	200 uL
	VSTD200	100 uL	100 uL	80 uL	400 uL

The internal standard is spiked by the autosampler. Due to different spike amounts separate standards are used depending on which autosampler is being used.

After analysis of the six points, the standard analyses must be quantitated and evaluated for adherence to QC criteria, as follows. Minimum requirements for method files are use of specific quantitation ions and quantitating a specific set of target compound and surrogates with a specified internal standard. These requirements are found in Tables 3 and 5.

7.4.4 Initial Calibration Criteria

The percent (%) RSD for six calibration check compounds (CCC) must be less than or equal to 30%. CCCs are 1,1-Dichloroethene, Chloroform, 1,2-Dichloropropane, Toluene, Ethylbenzene, and Vinyl Chloride.

A system performance check must be performed as part of initial calibration. The five system performance check compounds (SPCC) and the minimum acceptable average relative response factors (RRF) for these compounds are as follows (taken from 8260B):

SPCC	RRF
Chloromethane	0.10
1,1-Dichloroethane	0.10
Bromoform	0.10
Chlorobenzene	0.30
1,1,2,2-Tetrachloroethane	0.30

The SPCCs are used to check both the standard and instrument stability.

7.4.4.1 Linearity of Target Analytes

If the RSD of any target analyte is 15% or less using the average response factor, then the response factor is presumed to be constant

over the calibration range, and the average response factor may be used for quantitation.

If the RSD of any target analyte exceeds 15% using the average response factor, then a calibration option outlined in section 7.0 of method 8000 will need to be employed. Please note that some options may not be allowable for certain states, federal programs, or clients.

Option 1 (Section 7.5.2 of method 8000 - Rev. 2, 12/96), is a linear regression of instrument response versus the standard concentration. The correlation coefficient (r) for each target analyte and surrogate must be greater than or equal to 0.995. For linear models, Target calculates the correlation coefficient and then squares it (r^2). This is what is reported on all Target forms. The value for r^2 must be greater than or equal to 0.990.

Option 2 (Section 7.5.3 of method 8000 - Rev. 2, 12/96), is a non-linear calibration model not to exceed a third order (seven calibration points required) polynomial. The lab would use a quadratic model or second order polynomial. The use of a quadratic model requires six calibration points. In order for the quadratic model to be acceptable, the coefficient of determination must be greater than or equal to 0.99.

7.4.5 Independent Calibration Verification

Immediately following an initial calibration, an independent calibration standard must be analyzed. This standard contains all target compounds, internal standards and surrogates at a concentration of 50 ug/L and is obtained from a source independent of the initial calibration source. Please refer to section 8.1 and Table 1 for acceptance criteria and corrective action for this standard.
For projects or clients requiring DoD QSM 4.1 all project analytes must fall between 80-120% of the true value. No samples may be run until the ICV criteria are met.

7.4.6 Calibration Verification

Once a valid initial calibration curve has been achieved, a continuing calibration standard containing all the target compounds, internal standards and surrogates at a concentration of 50 ppb must be analyzed every 12-hour clock for Method 8260, timed from the injection of BFB. The relative response factor from the 50 ppb continuing calibration check standard must be compared to the average response factor data from the initial calibration.

The EICP (extracted ion current profile) area for any of the internal standards in the calibration verification must not change by more than a factor of two (-50% to +100%) from the same level standard in the last initial calibration. The retention time for any internal standard cannot shift by more than 30 seconds from the same level standard in the last initial calibration.

For Method 8260, if the percent difference for each CCC is less than or equal to 20%, and all of the SPCCs have a relative response factor greater than or equal to those listed in Section 7.4.3, the continuing calibration is considered valid.

For projects or clients requiring DoD QSM 4.1 all project analytes must have $\pm$ 20%D.

Continuing calibration check criteria must be met before sample analysis can proceed.

7.4.7 Retention Time Windows

Retention time windows are set at the midpoint standard of the calibration curve, following every ICAL. When a CV is analyzed (and not an ICAL), the retention time windows of the daily CV must be within 30 seconds of the midpoint calibration standard of the most recent ICAL. The samples analyzed following the daily CV must have retention times within 30 seconds of those for the daily CV. Each successive daily CV must be compared to the most recent ICAL midpoint standard.

For projects or clients requiring DoD QSM 4.1, IS responses and retention time windows for QC and samples are compared to the midpoint of the most recent ICAL.

7.5 QUALITY CONTROL SAMPLE ANALYSIS

When preparing standards in water or spiking samples with internal standards/surrogates or matrix spike solution, be sure to rinse all syringes a minimum of three times with purge and trap grade methanol between uses. Failure to do this will result in cross-contamination of samples and standards.

7.5.1 Laboratory Control Sample (LCS)

The LCS mix is prepared from a secondary source vendor (i.e. different vendor from the calibration standards). The LCS is analyzed immediately after the initial calibration curve or calibration check and prior to the method blank to minimize any analyte carryover possibilities in samples. Acceptance criteria for the LCS are outlined in Section 8.0.

To prepare the water and medium-level soil LCS, 25 uL of the LCS standard mix at 200 ug/mL are spiked into 100 mL of analyte-free laboratory reagent grade water for a final concentration of 50 ug/L. The Archon autosampler adds 1 uL of internal and 1 uL of surrogate standard to a 5 mL aliquot of this preparation for analysis. The Centurion autosampler adds 5 uL of both surrogates and internal standards to a 5 mL aliquot. To prepare the low-level soil LCS, a stir bar is added to 5 mL of the above solution in a VOA vial. The Archon unit adds an additional 10 mL of water to which the internal and surrogate standards have been added; this preparation is then heated, stirred and purged.

To prepare the water and medium-level soil LCS for analysis on the LSC 2000 / 2016 autosampler, 1.25 uL of the LCS standard mix at 200 ug/mL are spiked into 5 mL of analyte-free laboratory reagent grade water for a final concentration of 50 ug/L.

In the event that the batch MS/MSD requirement cannot be fulfilled, a laboratory Control Spike Duplicate must be analyzed.

7.5.2 Method Blank Analysis

After calibration criteria have been met, a method blank must be analyzed before sample analysis can proceed. A method blank analysis must be performed once for each 12-hour calibration immediately after analysis of the calibration standard(s) and prior to sample analysis.

The aqueous method blank is a volume of analyte free laboratory reagent grade water spiked with internal and surrogate standards.

The low-level soil method blank is a volume of analyte free laboratory reagent grade water spiked with internal and surrogate standards. This method blank is analyzed using the low soil specification.

The method blank must contain less than the Practical Quantitation Level (PQL) for all analytes of interest for the samples associated with the blank.

For projects requiring DoD QSM 4.1 no analytes may be detected >1/2 the PQL and > than the $1/10^{th}$ the measured amount in any sample or $1/10^{th}$ the regulatory limit, whichever is larger. Except for common laboratory contaminants which may not be detected > than the PQL.

7.5.3 Surrogate Recovery Limits

Laboratory established limits are derived for each of the surrogates. Please refer to the current revision of Katahdin Analytical Services SOP # QA-808 for further information on statistical limits. All samples including blanks, laboratory

control samples, matrix spikes and client samples, must meet the statistical limits for the analysis to be considered valid. If surrogate recoveries do not meet these limits, reanalysis must occur to confirm matrix interference.

7.5.4 Internal Standard Area Recoveries / Retention Times.

The internal standard responses and retention times in the method blank must be evaluated immediately after or during data acquisition. If the EICP (extracted ion current profile) area for any of the internal standard changes by a factor of two (-50% to +100%), from the last daily calibration standard, the GC/MS must by inspected, and corrective action taken. If the retention time for any internal standard has shifted by more than 30 seconds from the mid-point standard level of the most recent calibration sequence, the GC/MS must be inspected, and corrective action taken. All samples and QC must also meet the EICP area and retention time criteria or must be reanalyzed.

7.5.5 Matrix Spike/Matrix Spike Duplicate (MS/MSD) Analysis

An MS/MSD must be analyzed every twenty samples of a similar matrix. The MS/MSD is prepared in a manner similar to the LCS, except that 40 mL aliquots (aqueous) or 5 g aliquots (soil), of environmental samples are used in place of the analyte-free laboratory reagent grade water. Note that trip blanks and field/equipment blanks should not be used for MS/MSD analyses. The spike solution (section 7.5.1) is added to the sample at a concentration of 50 ppb. Acceptance criteria for the MS/MSD are outlined in Section 8.0.

In the event that sufficient volume of sample is not supplied to the laboratory so that an MS/MSD set cannot be analyzed within a batch of 20 samples, a laboratory control spike duplicate must be analyzed.

7.6 SAMPLE ANALYSIS

When new samples are received, they should be checked for past sample history. If sample history cannot be located or the sites are different than past sites, the project manager should be consulted. He/she may be able to provide more information about the sample. Sample history is used to determine what order in which to run the samples and at what dilution. Refer to Katahdin Analytical Services SOPCA-106, “Basic Laboratory Technique”, current revision for information on subsampling.

Samples are removed from the VOA refrigerator and appropriate chain of custody form is completed. Remove only the vials that have not been opened yet (opened vials will be upside down). Note in sample run log any bubbles, and significant discoloration or sediment in the sample vials.

7.6.1 SAMPLE ANALYSIS FOR 8260B WATER

7.6.1.1 Tekmar LSC 2000 / 2016 units

Rinse a 5.0 mL gas-tight syringe a minimum of three times with analyte-free laboratory reagent grade water (e.g., Poland Spring or equivalent). Pour sample at ambient temperature into the syringe until nearly overflowing. Carefully insert and adjust plunger to sample volume of 5.0 mL. While adjusting plunger to final volume, expel extra volume of sample onto pH paper for sample pH verification. Add 1.0 uL of the internal and surrogate mixtures (250 ug/mL). Immediately inject contents of the syringe into the ALS sparger.

Record the sample pH in the injection logbook. Continue as above for each sample, ensuring that the 5.0 mL gas-tight syringe is rinsed a minimum of three times with laboratory reagent grade water between each sample.

7.6.1.2 Tekmar LSC 3000 / Archon 5100 units

Place the sample vials into the Archon sample tray and program the Archon for the appropriate sample volume and or dilution for the sample. The Archon unit will automatically transfer the sample to the sparge vessel while adding the internal and surrogate standard. The Archon can be programmed to run as many samples as will fit in the twelve-hour window. The auto sampler hot water rinses the sparge vessel, transfer lines, purge needle, and syringe between samples to minimize possible carryover.

Record the sample pH in the injection logbook after sample analysis is complete (usually the day after the analysis is done) and return the sample vial to the sample refrigerator.

7.6.1.3 Centurion/Encon unit

Place the sample vials into the Centurion sample tray and program the Centurion for the proper sequence. The Centurion will automatically transfer the sample to the sparge vessel while adding the internal and surrogate standards. Using the Centurion software, the analyst can program the Centurion to run as many samples that will fit into a 12 hour clock. The autosampler uses hot water to rinse the sparge vessel, transfer lines, purge needle and sample needle to minimize carryover.

Record the sample pH in the injection logbook after sample analysis is complete (usually the day after the analysis is done) and return the sample vial to the sample refrigerator.

Make sure that all entries in the injection log have been made in a complete, neat, and legible manner. Corrections in any logbook must be crossed through with a single line, dated, initialed and have a written explanation or the applicable error code.

If for any reason a sample needs to be rerun, diluted or duplicated, a note in the comments field of the injection logbook must be entered, addressing the reason why in the logbook to facilitate answering any questions that may arise during the review process.

To minimize carryover from samples that contain a target compound at a level exceeding the upper limit of the calibration curve, the following must be done: monitor both the samples immediately after the contaminated sample as well as the next run of the contaminated sample in the same purge inlet for the target(s) in question; both must have levels <PQL.

7.6.2 ANALYSIS OF LOW-LEVEL SOIL SAMPLES

Method 5035 Closed System Purge & Trap procedure for low level soils (5 ug/Kg -200 ug/Kg)

Selecting the appropriate technique may depend on cleanup goals, confidence levels, and anticipated levels of contamination. Field sampling activities typically result in Encore or Encore-like devices being submitted to the lab. These devices must be extruded within 48 hours. It is the laboratory's standard policy to extrude soil samples into 5 mL of Laboratory reagent free laboratory reagent grade water that contains a magnetic stir bar. The sample is subsequently frozen until analysis within 14 days. Note that the sample must be extruded and frozen within 48 hours of sampling, until analysis can begin. This approach is preferred over extrusion into sodium bisulfate because it is believed that the sodium bisulfate reacts with calcium carbonate in highly calcareous soils causing effervescence and driving the volatile analytes out of solution. There is also anecdotal information to suggest that acetone may be generated when bisulfate preservation occurs. The Katahdin sample ID, extrusion date, and time are recorded in the GC/MS extrusion logbook. Please refer to the Katahdin method 5035 SOP, CA-214 for more detail.

In lieu of the use of Encore samplers, the lab may pre-weigh 40 mL VOA vials containing 5 mL of laboratory reagent grade water or a 20% sodium bisulfate solution and a magnetic stir bar and ship these to the field. The vial

is assigned a vial specific number prior to shipment to the field. The vial and weight will be recorded with its vial specific number in the methanol soil logbook. If possible the field sampler should weigh the sealed vial to ensure that 5 +/- 0.5 grams of sample were added in the field. When the lab receives the vials back from the field, the vials will be weighed and the weight recorded. The samples must be frozen within 48 hours of sampling, until analysis can begin.

The subsequent analysis is performed on a specially developed autosampler that heats, stirs, and purges the sample simultaneously without exposing the contents of the vial to the atmosphere. This procedure will help to minimize the loss of VOC's due to transport, handling, and analysis and may help minimize ambient lab contribution. The expected detection limits are consistent with the traditional low soil technique from method 5030. The Archon is programmed to heat each vial to 40°C during the purge time. Initiate purging for 11.0 minutes; the sample must be heated to 40°C ± 1°C before purging can begin. If you have questions concerning setting up the Tekmar or initiating a GC/MS batch run, consult the Organic Department Manager, or senior chemist within the group.

If the client does not require method 5035, method 5030 for analysis of low-level soils may be followed. This means that the Tekmar ALS 2016 unit may be used for the preparative step, as well as the Archon units.

7.6.3 ANALYSIS OF MEDIUM-LEVEL SOIL SAMPLES

Method 5030 Procedure for higher concentration soils (> 200 ug/Kg)

Higher concentration soils may be sampled as either a bulk sample or field preserved with a water miscible solvent such as methanol. If sampled in an Encore unit, the soil is extruded into methanol upon receipt at the lab.

Bulk Sample- A sample is placed in a glass jar or vial and returned to the lab for extraction and analysis. In this approach the lab takes an aliquot of soil and extracts with purge & trap grade methanol, a portion of the methanol is then analyzed for volatile analytes.

Extraction

Calibrate the balance properly (See SOP CA-102) and note it in the appropriate logbook. Place 5.0 grams of thoroughly mixed, undecanted soil sample in a 40.0 mL vial. Add 5.0 mL reagent grade methanol. Shake for 2 minutes. Let stand for 3 minutes. Record extraction in soil prep logbook.

Methanol Field Preservation - A 5 gram sample is added to a VOA vial that has been previously charged with purge and trap grade methanol (the

volume of methanol is dependent upon client request). The vial with methanol has been previously weighed in the lab and assigned a vial specific number prior to shipment to the field. The vial and methanol weight will be recorded with its vial specific number in the VOA vial prep logbook. If possible the field sampler should weigh the sealed vial to ensure that 5 +/- 0.5 grams of sample were added in the field. When the lab receives the vials back from the field, the vials will be weighed and the weight recorded. A portion of the methanol is then analyzed for volatile analytes.

For analysis on Archon or Centurion autosamplers, add 400 uL of the extract into 20 mL of organic-free laboratory reagent grade water (e.g., Poland Spring or equivalent). IS and SS is added by the Archon and/or Centurion autosampler for analysis. This will give an estimated calibration range between 500-10000 ug/Kg.

7.7 FINAL DATA PACKAGE

7.7.1 Initial Data Review (IDR)

The initial data review is performed by the analyst who ran the samples. This review is of sufficient quality and detail to provide a list of samples that need to be reanalyzed or diluted and reanalyzed. The initial data review is performed on the detailed quantitation reports of the analyzed sample. This data review examines criteria that directly impact whether or not the sample needs to be reanalyzed.

- Surrogate recoveries
- stability of internal standard responses
- LCS spike recoveries
- method blank acceptance
- chromatography
- target compound detection/quantitation / review for false positives

The analyst must evaluate all data using the QA Acceptance Criteria table found within this SOP (Table 1). This table gives acceptance criteria and corrective actions for criteria that are not met. In addition to evaluating QC elements, the chromatography and quantitation of target analytes must be reviewed.

7.7.1.1 Chromatography

The chromatography should be examined for the presence or absence of any "ghost" peaks and can also be used as an indication of whether or not matrix interferences might be influencing surrogate recoveries and/or ISTD area recoveries. Whether or not the chromatography is

acceptable is a judgment call on the part of the analyst and should be used in conjunction with other monitored QC (e.g., Surrogate recoveries) to determine the necessity of reanalyses.

Manual integrations are to be performed when chromatographic conditions preclude the computer algorithm from correctly integrating the peak of concern. In no instance shall a manual integration be performed solely to bring a peak within criteria.

Each peak of concern is examined by the primary analyst to ensure that the peak was integrated properly by the computer algorithm. An "M" qualifier will automatically be printed on the quantitation report summary.
This manual integration package must then be submitted to the Organic Department Manager or his/her designee, who will review each manual integration.

For specific procedures on how to manually integrate, refer to Katahdin SOP QA-812, "Manual Integration", current revision.

7.7.1.2 Target Compound Detection/Quantitation

The method files have been set up to error on the side of false positives, that is to identify and quantitate peaks as target compounds that may not necessarily be valid hits.

The requirements for qualitative verification by comparison of mass spectra are as follows:

- all ions present in the standard mass spectra at a relative intensity > 25% must be present in the sample spectrum.
- the relative intensities of primary and secondary ions must agree within ±20% between the standard and sample spectra.
- ions greater than 25% in the sample spectrum but not present in the standard spectrum must be considered and accounted for by the analyst.

If a compound cannot be verified by all three criteria above, but, in the technical judgment of the mass spectral interpretation specialist, the identification is correct, then the laboratory shall report that compound on the Form 1 as a valid hit.

If any target concentration exceeds the upper limit, a dilution must be made and analyzed. The dilution chosen should keep the response of the largest target compound hit in the upper half of the initial calibration range.

The GC/MS laboratory initial data review must be completed within twelve hours of batch completion; in the majority of instances, the initial data review should be accomplished at the beginning of a work shift for the previous set of analyses. After the analyst has completed his or her initial data review, the data should immediately be forwarded to the Organic Department Manager, or his/her designee.

7.7.1.3 Tentatively Identified Compounds (TIC)

TIC's may be requested by certain clients for samples. Refer to SOP CA-207 "GC/MS Library Search and Quantitation".

7.7.2 Reporting

After the chromatograms have been reviewed and any target analytes have been quantitated using Target, the necessary files are brought into QuickForms. Depending on the QC level requested by the client, a Report of Analysis (ROA) and additional reports, such as LCS forms and chronology forms, are generated. The package is assembled to include the necessary forms and raw data. The data package is reviewed by the primary analyst and then forwarded to the secondary reviewer. The secondary reviewer validates the data and checks the package for any errors. When completed, the package is sent to the department manager for final review. A completed review checklist is provided with each package. The final data package from the Organics department is then processed by the Data Management department.

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

Refer to Table 1 and to details in this section for a summary of QC requirements, acceptance criteria, and corrective actions. These criteria are intended to be guidelines for analysts. The criteria does not cover all possible situations. If any of the QC requirements are outside the recovery ranges listed in this section or in Table 1, all associated samples must be evaluated against all the QC. In some cases data may be reported, but may be reanalyzed in other cases. Making new reagents and standards may be necessary if the standardization is suspect. The corrective actions listed in this section and in Table 1 may rely on analyst experience to make sound scientific judgments. These decisions are based on holding time considerations, client and project specific Data Quality Objectives and on review of chromatograms. The Department Manager, Operations Manager, and/or Quality Assurance Officer may be consulted to evaluate data. Some samples may not be able to be reanalyzed within hold time. In these cases "qualified" data with narration may be advisable after consultation with the client.

In some cases the standard QC requirements listed in this section and in Table 1 may not be sufficient to meet the Data Quality Objectives of the specific project. Much of the work

performed at the lab is analyzed in accordance with specific QC requirements spelled out in a project specific Quality Assurance Project Plan (QAPP) or in a program specific Quality Systems Manual (QSM). The reporting limits, acceptance criteria and/or corrective actions may be different than those specified in this SOP. In these cases the appropriate information will be communicated to the Department Manager and/or senior chemists before initiation of the analyses so that specific product codes can be produced for the project. In addition, the work order notes for each project will describe the specific QAPP or QSM to be followed.

8.1 Independent Calibration Verification, LCS and MS/MSD Criteria

Statistical limits are compiled annually for LCS recoveries (archived in QA office). Statistical limits are only calculated when at least 30 usable data points are obtained for any given compound. If insufficient data points are available, nominal limits are set by the Organic Department Manager, Laboratory Operations Manager and Quality Assurance Officer. Refer to Katahdin SOP QA-808, "Generation and Implementation of Statistical QC Limits and/or Control Charts," current revision.

The use of statistical limits versus nominal limits is dependent on the client and project. This information is communicated to the Organic Department Manager through the Katahdin project manager. It is standard practice to use statistical limits for reporting purposes and to evaluate any QC criteria exceedances. However, nominal limits of 60-140% or 70-130% may be used for some projects or states.

The LCS recoveries for all analytes are evaluated. All of the compounds of interest must fall within the established statistical limits with the following sporadic exceedance allowances.

Number of Analytes	Number of Allowable Exceedances
> 90	5
71 – 90	4
51 – 70	3
31 – 50	2
11 – 30	1
<11	0

If less than the number of allowable exceedances fail the statistical limits, no corrective action is needed. If greater than the number of allowable exceedances fail the statistical limits, corrective action may be taken. Corrective actions may vary with each situation. However, in the case where the failures are high and the samples are non-detect for those compounds, then no corrective action is required. Otherwise, corrective action may involve reanalysis or recalibration. The specific corrective actions taken will rely on analyst experience to make sound scientific judgments while considering client objectives, other quality control indicators and/or the ability to reanalyze a sample within holding time.

The MS/MSD recoveries for all analytes are evaluated. If the LCS results are acceptable but the MS/MSD is not, narrate. If both the LCS and MS/MSD are unacceptable reprep the samples and QC.

Please note that for compounds with only nominal limits (i.e. insufficient data points were available to generate statistical limits), no corrective action is required for out-of-criteria recoveries until enough data points are established to generate statistical limits.

For projects or clients requiring DoD QSM 4.1 all project analytes in the ICV must fall between 80-120% of the true value. No samples may be run until the ICV criteria is met. Laboratory established recovery limits for LCS and MS/MSDs must be within 3 standard deviations of the mean LCS recovery. MS/MSD pairs must be run once per analytical/preparatory batch. RPDs must be less than or equal to 30% between MS and MSDs.

For analytes with no available DoD acceptance criteria, laboratory established limits shall be used.

8.2 Surrogate Recovery Criteria

Statistical limits are compiled annually for surrogate recoveries (archived in QA office). Statistical limits are only calculated when at least 30 usable data points are obtained for any given compound. If insufficient data points are available, nominal limits are set by the Organic Department Manager, Laboratory Operations Manager and Quality Assurance Officer. The use of statistical limits versus nominal limits is dependent on the client and project. This information is communicated to the Organic Department Manager through the Katahdin project manager. It is standard practice to use statistical limits for reporting purposes and to evaluate any QC criteria exceedances. However, nominal limits of 60-140% or 70-130% may be used for some projects or states.

8.3 QC Requirements

Refer to Table 1 for a summary of QC requirements, acceptance criteria, and corrective actions. Table 1 criteria are intended to be guidelines for analysts. The table does not cover all possible situations. If any of the QC requirements are outside the recovery ranges listed in Table 1, all associated samples must be evaluated against all the QC. In some cases data may be reported, but may be reanalyzed in other cases. Making new reagents and standards may be necessary if the standardization is suspect. The corrective actions listed in Table 1 may rely on analyst experience to make sound scientific judgments. These decisions are based on holding time considerations, client and project specific Data Quality Objectives and on review of chromatograms. The Department Manager, Operations Manager, and/or Quality Assurance Officer may be consulted to evaluate data. Due to the 14-day hold time associated with this method, samples may not be able to be reanalyzed

within hold time. In these cases "qualified" data with narration may be advisable after consultation with the client.

9.0 METHOD PERFORMANCE

The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero. The MDLs are determined annually per type of instrument and filed with the Organic Department Manager and with the QAO. Refer to the current revision of Katahdin SOP QA-806, Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications, for procedures on determining the MDL.

Refer to the current revision of Method 8260 for other method performance parameters and requirements.

10.0 APPLICABLE DOCUMENTS/REFERENCES

Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, USEPA SW846, 3rd Edition, Final Updates I, II, IIA, IIB, III, IIIA, IIIB and IV, February 2007, Method 8260B.

"Department of Defense Quality Systems Manual for Environmental Laboratories" (DoD QSM), Version 4.1, 04/22/09.

"The National Environmental Laboratory Accreditation Conference (NELAC) Standards," June 2003.

Katahdin SOP CA-101, Equipment Maintenance and Troubleshooting, current revision.

Katahdin SOP QA-806, Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications, current revision.

LIST OF TABLES AND FIGURES

TABLE 1

QC REQUIREMENTS - VOLATILE ORGANICS, METHOD 8260

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Check of mass spectral ion intensities using BFB	Prior to initial calibration and calibration verification	Refer to the criteria listed in Section 7.3 of this SOP	Retune instrument, and verify
Six-point calibration for all analytes	Initial calibration prior to sample analysis	SPCCs average RF ≥0.30, except chloromethane, 1,1-DCA and bromoform ≥0.10; RSD for RFs ≤ 30% for CCCs. Refer to section 7.4.3 also.	Repeat initial calibration
Independent Calibration Verification	Once, immediately following calibration	Statistically derived from lab data or nominal limits depending on the project. Refer to QA records for statistical limits. Nominal limits are used as default limits. See also section 8.1 of this SOP for more information on allowable exceedances	Evaluate the samples and associated QC: i.e. If an MS/MSD was performed and acceptable, narrate. If an LCS/LCSD was performed and only one of the set was unacceptable, narrate. If the surrogate recoveries in the LCS are also low but are acceptable in the blank and samples, narrate. If the LCS recovery is high but the sample results are <PQL, narrate. Otherwise, reprep a blank and the remaining samples.
Calibration verification	Once per each 12 hours, prior to sample analysis in absence of initial cal	SPCCs minimum RF ≥ 0.30, except chloromethane, 1,1-DCA and bromoform ≥ 0.10; RF for CCC analytes ≤ 20% (%D) of average initial multipoint RF	Repeat initial calibration and reanalyze all samples analyzed since the last successful calibration verification
IS	During data acquisition of calibration check standard	Retention time ± 30 seconds; EICP area within -50% to +100% of last calibration verification (12 hours) for each IS	Inspect mass spectrometer or GC for malfunctions; mandatory reanalysis of samples analyzed while system was malfunctioning
Method Blank	One per batch of 20 or fewer samples.	No analytes of interest detected > PQL with the exception of Methylene Chloride	(1) Investigate source of contamination (2) Evaluate the samples and associated QC: i.e. If the blank results are above the PQL, report sample results which are <PQL or > 10X the blank concentration. Otherwise, reprep a blank and the remaining samples.

TABLE 1 (cont.)

QC REQUIREMENTS - VOLATILE ORGANICS, METHOD 8260

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
LCS	One per batch of 20 or fewer samples.	Statistically derived from lab data or nominal limits depending on the project. Refer to QA records for statistical limits. Nominal limits are used as default limits. See also section 8.4 of this SOP for more information on allowable exceedances. .	Evaluate the samples and associated QC: i.e. If an MS/MSD was performed and acceptable, narrate. If an LCS/LCSD was performed and only one of the set was unacceptable, narrate. If the surrogate recoveries in the LCS are also low but are acceptable in the blank and samples, narrate. If the LCS recovery is high but the sample results are <PQL, narrate. Otherwise, reprep a blank and the remaining samples.
Surrogate spike	Every sample, control, standard and method blank	Statistically derived limits.	Reprep and reanalyze for confirmation of matrix interference when appropriate.
MS/MSD	One MS/MSD per every 20 samples.	Statistically derived from lab data or nominal limits depending on the project. Statistical limits are used as default limits. .	(1) Evaluate the samples and associated QC: i.e. If the LCS results are acceptable, narrate. (2) If both the LCS and MS/MSD are unacceptable reprep the samples and QC.
MDL Study	Refer to KAS SOP QA-806, "Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications", current revision.		
Demonstrate ability to generate acceptable P & A using 4 replicate analyses of a QC check standard	Once per year for each analyst; 4 reps	All recoveries within method QC acceptance limits	Recalculate results; locate and fix problem; rerun P & A study for those analytes that did not meet criteria prior to sample analysis

TITLE: **ANALYSIS OF VOAs BY PURGE AND TRAP GC/MS: SW-846 METHOD 8260**

TABLE 2

DOD QSM Version 4.1 QC Requirements

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Demonstrate acceptable analytical capability	Prior to using any test method and at any time there is a significant change in instrument type, personnel, test method, or sample matrix.	QC acceptance criteria published by DoD, if available; otherwise, method-specific criteria.	Recalculate results; locate and fix problem, then rerun demonstration for those analytes that did not meet criteria.	NA.	This is a demonstration of analytical ability to generate acceptable precision and bias per the procedure in Appendix C. No analysis shall be allowed by analyst until successful demonstration of capability is complete.
LOD determination and verification	Refer to current revision of SOP QA-806				
LOQ establishment and verification	Refer to current revision of SOP QA-806				
Tuning	Prior to ICAL and at the beginning of each 12-hour period.	Refer to method for specific ion criteria.	Retune instrument and verify. Rerun affected samples.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be accepted without a valid tune.
Minimum five-point initial calibration (ICAL) for all analytes	ICAL prior to sample analysis.	1. Average response factor (RF) for SPCCs: VOCs ≥ 0.30 for chlorobenzene and 1,1,2,2-tetrachlorolethane; ≥ 0.1 for chloromethane, bromoform, and 1,1-dichloroethane. 2. RSD for RFs for CCCs ≤ 30% and one option below: Option 1: RSD for each analyte ≤ 15%; Option 2: linear least squares regression r ≥ 0.995; Option 3: non-linear regression–coefficient of determination (COD) r2 ≥ 0.99 (6 points shall be used for second order).	Correct problem then repeat ICAL.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be run until ICAL has passed. Calibration may not be forced through the origin.
Second source calibration verification (ICV)	Once after each ICAL.	All project analytes within ± 20% of true value.	Correct problem and verify second source standard. Rerun second source verification. If that fails, correct problem and repeat ICAL.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be run until calibration has been verified.

TABLE 2 (cont)

DOD QSM Version 4.1 QC Requirements

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Retention time window position establishment for each analyte and surrogate	Once per ICAL.	Position shall be set using the midpoint standard of the ICAL curve when ICAL is performed. On days when ICAL is not performed, the initial CCV is used.	NA.	NA.	
Evaluation of relative retention times (RRT)	With each sample.	RRT of each target analyte within ± 0.06 RRT units.	Correct problem, then rerun ICAL.	Flagging criteria are not appropriate.	Laboratories may update the retention times based on the CCV to account for minor performance fluctuations or after routine system maintenance (such as column clipping). With each sample, the RRT shall be compared with the most recently updated RRT. If the RRT has changed by more than ±0.06 RRT units since the last update, this indicates a significant change in system performance and the laboratory must take appropriate corrective actions as required by the method and rerun the ICAL to reestablish the retention times.
Continuing calibration verification (CCV)	Daily before sample analysis and every 12 hours of analysis time.	1. Average RF for SPCCs ≥ 0.30 for chlorobenzene and 1,1,2,2-tetrachlorolethane; ≥ 0.1 for chloromethane, bromoform, and 1,1-dichloroethane. 2. %Difference/Drift for all target compounds and surrogates ≤ 20%D (Note: D = difference when using RFs or drift when using least squares regression or non-linear calibration).	DoD project level approval must be obtained for each of the failed analytes or corrective action must be taken. Correct problem, then rerun calibration verification. If that fails, then repeat ICAL. Reanalyze all samples since last acceptable CCV.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to all results for the specific analyte(s) in all samples since last acceptable CCV.	Problem must be corrected. Results may not be reported without a valid CCV. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Internal standards verification	Every field sample, standard, and QC sample.	Retention time ± 30 seconds from retention time of the midpoint standard in the ICAL; EICP area within -50% to +100% of ICAL midpoint standard.	Inspect mass spectrometer and GC for malfunctions. Reanalysis of samples analyzed while system was malfunctioning is mandatory.	If corrective action fails in field samples, apply Q-flag to analytes associated with the non-compliant IS. Flagging criteria are not appropriate for failed standards.	Sample results are not acceptable without a valid IS verification.

TABLE 2 (cont)

DOD QSM Version 4.1 QC Requirements

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Method blank	One per preparatory batch.	No analytes detected > ½ RL (> RL for common lab contaminants) and > 1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater). Blank result must not otherwise affect sample results.	Correct the problem. Report sample results that are <LOD or >10x the blank concentration. Reprepare and reanalyze the method blank and all associated samples with results > LOD and < 10x the contaminated blank result. Contact Client if samples cannot be reprepped within hold time.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply B-flag to all results for the specific analyte(s) in all samples in the associated preparatory batch.	Problem must be corrected. Results may not be reported without a valid method blank. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
LCS containing all analytes to be reported, including surrogates	One per preparatory batch.	The laboratory shall use laboratory control limits (CLs) or use DoD-generated LCS-CLs, if available depending on project requirements. In-house CLs may not be greater than ± 3 times the standard deviation of the mean LCS recovery. A number of analytes may fall outside the CL but within marginal exceedance limit depending on the total number of analytes in the LCS.	Correct problem, then reprep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available. Refer to Table G-1 for number of marginal exceedences allowed. Contact Client if samples cannot be reprepped within hold time.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch.	Problem must be corrected. Results may not be reported without a valid LCS. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Matrix Spike (MS)	One per preparatory batch per matrix if sufficient sample is available.	For matrix evaluation, use LCS acceptance criteria.	Examine the project-specific DQOs. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met.	For matrix evaluation only. If MS results are outside the LCS limits, the data shall be evaluated to determine the source of difference and to determine if there is a matrix effect or analytical error.
Matrix spike duplicate (MSD) or sample duplicate	One per preparatory batch per matrix if sufficient sample is available.	MSD: For matrix evaluation, use LCS acceptance criteria. MS/MSD: RPD ≤ 30% .	Examine the project-specific DQOs. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met.	The data shall be evaluated to determine the source of difference.

TABLE 2 (cont)

DOD QSM Version 4.1 QC Requirements

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Surrogate spike	All field and QC samples.	The laboratory shall use laboratory surrogate CLs or use DoD-generated surrogate CLs, if available depending on project requirements. .	For QC and field samples, correct problem then reprep and reanalyze all failed samples for failed surrogates in the associated preparatory batch, if sufficient sample material is available. If obvious chromatographic interference with surrogate is present, reanalysis may not be necessary. Contact Client if samples cannot be reprepped within hold time.	Apply Q-flag to all associated analytes if acceptance criteria are not met.	Alternative surrogates are recommended when there is obvious chromatographic interference.
Results reported between DL and LOQ	NA.	NA.	NA.	Apply J-flag to all results between DL and LOQ.	

TITLE: **ANALYSIS OF VOAs BY PURGE AND TRAP GC/MS: SW-846 METHOD 8260**

TABLE 3

SUMMARY OF METHOD MODIFICATIONS

TOPIC	KATAHDIN SOP CA-202-11	METHOD 8260, current revision
Apparatus/Materials	None	
Reagents	None	
Sample preservation/ handling	Preserved samples analyzed within 14 days. Unpreserved samples analyzed within 7 days.	Preserved samples analyzed within 14 days. No criteria for unpreserved samples.
Procedures	(1) Use laboratory reagent grade water for low level soil calibration, method blanks, and laboratory control samples to minimize clogging of archon soil needles with sand. (2) Internal Standards- pentafluoro-benzene, 1,4-difluorobenzene, chlorobenzene-d5, 1,4-dichloro-benzene-d4	(1) Use an aliquot of a clean (control) matrix similar to the sample matrix. (2) Recommended internal standards – fluorobenzene, chlorobenzene-d5, 1,4-dichloro-benzene-d4
QC - Spikes	None	
QC - LCS	None	
QC - Accuracy/Precision	PQL – Practical Quantitation Level – three to ten times the MDL.	EQL – Estimated Quantitation Level – five to ten times the MDL
QC - MDL	None	

TABLE 4

VOA COMPOUNDS AND CHARACTERISTIC IONS

COMPOUND	1° ION	2° ION
Acetone	43	58
Acetonitrile	41	40, 39
Acrolein	56	55, 58
Acrylonitrile	53	52, 51
Allyl Chloride	76	41, 39
Benzene	78	-
Bromobenzene	156	77, 158
Bromochloromethane	128	49, 130
Bromodichloromethane	83	85, 127
Bromoform	173	175, 254
Bromomethane	94	96
2-Butanone	43	72
n-Butylbenzene	91	92, 134
Sec-Butylbenzene	105	134
Tert-Butylbenzene	119	91, 134
Carbon Disulfide	76	78
Carbon Tetrachloride	117	119
Chlorobenzene	112	77, 114
Chloroethane	64	66
2-Chloroethylvinyl Ether	63	65, 106
Chloroform	83	85
Chloromethane	50	52
Chloroprene	53	88, 90
2-Chlorotoluene	91	126
4-Chlorotoluene	91	126
Cyclohexane	56	84, 60
1,2-Dibromo-3-Chloropropane	75	155, 157
Dibromochloromethane	129	127
1,2-Dibromoethane	107	109, 188
Dibromomethane	93	95, 174
Diethyl Ether	74	45, 59
1,2-Dichlorobenzene	146	111, 148
1,3-Dichlorobenzene	146	111, 148
1,4-Dichlorobenzene	146	111, 148
Dichlorodifluoromethane	85	87
1,1-Dichloroethane	63	65, 83
1,2-Dichloroethane	62	98
1,1-Dichloroethene	96	61, 63
Cis-1,2-Dichloroethene	96	61, 98
Trans-1,2-Dichloroethene	96	61, 98
1,2-Dichloropropane	63	112

TABLE 4 (cont.)

VOA COMPOUNDS AND CHARACTERISTIC IONS

COMPOUND	1° ION	2° ION
1,3-Dichloropropane	76	78
2,2-Dichloropropane	77	97
1,1-Dichloropropene	75	110, 77
Cis-1,3-Dichloropropene	75	77, 39
Trans-1,3-Dichloropropene	75	77, 39
Cis-1,4-Dichloro-2-butene	75	53, 77
Trans-1,4-Dichloro-2-butene	53	88, 75
1,4-Dioxane	88	58, 43
Di-Isopropyl Ether	45	43, 87
Ethylbezene	91	106
Ethyl Methacrylate	69	41, 99
Ethyl Tertiary-Butyl Ether	59	87, 57
Freon-113	151	101
Hexachlorobutadiene	225	223, 227
2-Hexanone	43	58, 57, 100
Idomethane	142	127, 141
Isobutyl Alcohol	43	41, 42
Isopropylbezene	105	120
P-ISOPROPYLTOLUENE	119	134, 91
Methacrylonitrile	41	67, 39
Methylcyclohexane	83	55, 98
Methylene Chloride	84	86, 49
Methyl Acetate	43	74
Methyl Methacrylate	69	41, 100
4-Methyl-2-Pentanone	43	58, 85, 100
Methyl Tert-Butyl Ether	73	57, 41
Naphthalene	128	-
Pentachloroethane	167	130, 132
Propionitrile	54	52, 55
N-PROPYLBENZENE	91	120
Styrene	104	78
Tertiary-Amyl Methyl Ether	73	55, 87, 71
Tertiary-Butyl Alcohol	59	41, 43
1,1,1,2-Tetrachloroethane	131	133, 119
1,1,2,2-Tetrachloroethane	83	131, 85
Tetrachloroethene	164	129, 131, 166
Tetrahydrofuran	42	72, 71
Toluene	92	91
1,2,3-Trichlorobenzene	180	182, 145
1,2,4-Trichlorobenzene	180	182, 145
1,3,5-Trichlorobenzene	180	182, 145
1,1,1-Trichloroethane	97	99, 61
1,1,2-Trichloroethane	83	97, 85
Trichloroethene	95	97, 130, 132

TABLE 4 (cont.)

VOA COMPOUNDS AND CHARACTERISTIC IONS

COMPOUND	1° ION	2° ION
Trichlorofluoromethane	151	101, 153
1,2,3-Trichloropropane	75	77
1,2,3-Trimethylbenzene	105	120
1,2,4-Trimethylbenzene	105	120
1,3,5-Trimethylbenzene	105	120
Vinyl Acetate	43	86
Vinyl Chloride	62	64
Xylenes (Total)	106	91
1-Chlorohexane	91	55,43

TITLE: **ANALYSIS OF VOAs BY PURGE AND TRAP GC/MS: SW-846 METHOD 8260**

TABLE 5

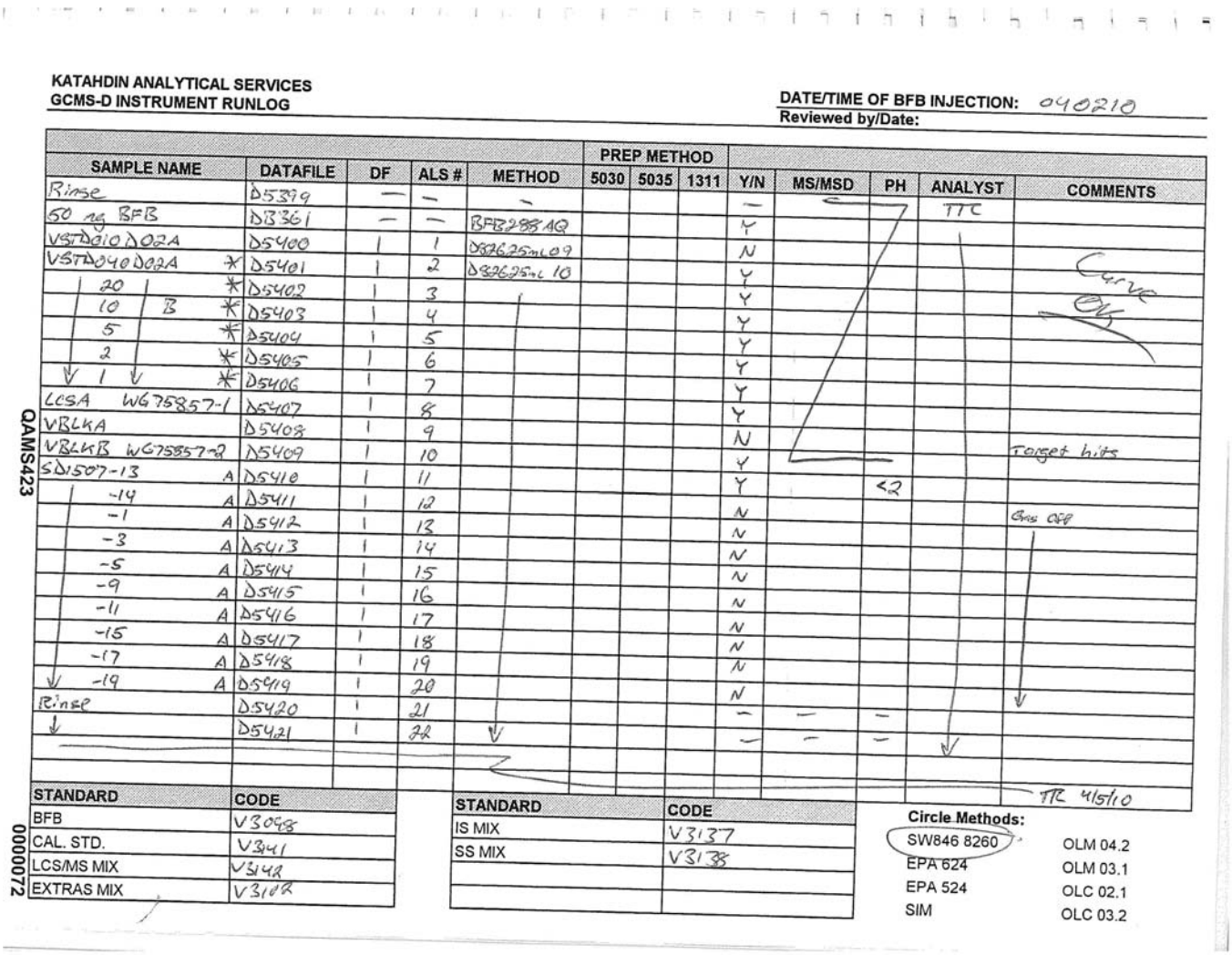
ANALYTE QUANTITATION AND INTERNAL STANDARDS

Pentafluorobenze	**1,4-Difluorobenzene**	**Chlorobenzene - d5**	**1,4-Dichlorobenzene - d4**
Dichlorodifluoromethane	1,2-Dichloroethane	1,3-Dichloropropane	1,1,2,2-Tetrachloroethane
Chloromethane	1,1-Dichloropropene	Tetrachloroethene	1,2,3-Trichloropropane
Bromomethane	Carbon tetrachloride	Dibromochloromethane	Isopropylbenzene
Vinyl chloride	Benzene	Chlorobenzene	Bromobenzene
Chloroethane	1,2-Dichloropropane	1,1,1,2-Tetrachloroethane	2-Chlorotoluene
Trichlorofluoromethane	Trichloroethene	Ethylbenzene	4-Chlorotoluene
Methylene Chloride	Dibromomethane	Xylenes (total)	1,3,5-Trimethylbenzene
Acetone	Bromodichloromethane	Bromoform	Tert-Butylbenzene
1,1-Dichloroethene	cis -1,3-Dichloropropene	Styrene	1,2,4-Trimethylbenzene
1,1-Dichloroethane	4-Methyl-2-pentanone	2-Hexanone	Sec-Butylbenzene
cis-1,2-Dichloroethene	Toluene-d8 (surr.)	Bromoform	1,3-Dichlorobenzene
trans-1,2-Dichloroethene	Toluene		P-Isopropyltoluene
Chloroform	trans-1,3-Dichloropropene		1,4-Dichlorobenzene
2,2-Dichloropropane	1,1,2-Trichloroethane		1,2-Dichlorobenzene
2-Butanone	1,2-Dibromoethane		N-Propylbenzene
Methyl-tert-butylether (MTBE)	Vinyl Acetate		1,2-Dibromo-3-chloropropane
Tetrahydrofuran	Methyl Methacrylate		1,2,4-Trichlorobenzene
Bromochloromethane	Ethyl Methacrylate		Naphthalene
1,1,1-Trichloroethane	1,4-Dioxane		Hexachlorobutadiene
Tertiary-butyl alcohol (TBA)	2-Chloroethylvinyl ether		1,2,3-Trichlorobenzene
Di-isopropyl ether (DIPE)	Bromofluorobenzene (surr.)		cis-1,4-Dichloro-2-butene
Ethyl-tert-butylether (ETBE)			trans-1,4-Dichloro-2-butene
Tertiary-amyl methyl ether			Pentachloroethane
Diethyl Ether			n-Butylbenzene
Carbon Disulfide			1,3,5-Trichlorobenzene
Freon-113			1,2,3-Trimethylbenzene
Iodomethane			
Acrolein			
Isobutyl Alcohol			
Allyl Chloride			
Chloroprene			
Propionitrile			
Methacrylonitrile			
Acrylonitrile			
Cyclohexane			
Methyl Acetate			
Methylcyclohexane			
1-Chlorohexane			
Dibromofluoromethane (surr.)			
1,2-Dichloroethane-d4 (surr.)			

TITLE: **ANALYSIS OF VOAs BY PURGE AND TRAP GC/MS: SW-846 METHOD 8260**

FIGURE 1

EXAMPLE OF VOA RUNLOG PAGE



KATAHDIN ANALYTICAL SERVICES
GCMS-D INSTRUMENT RUNLOG

DATE/TIME OF BFB INJECTION: 040210
Reviewed by/Date:

SAMPLE NAME	DATAFILE	DF	ALS #	METHOD	PREP METHOD 5030	5035	1311	Y/N	MS/MSD	PH	ANALYST	COMMENTS
Rinse	D5399	—	—	—				—			TTC	
50 ng BFB	D3361	—	—	BFB288AQ				Y				
VSTD010D02A	D5400	1	1	D826.25mL09				N				
VSTD040D03A *	D5401	1	2	D826.25mL10				Y				
20 *	D5402	1	3					Y				Curve
10 B *	D5403	1	4					Y				Ok
5 *	D5404	1	5					Y				
2 *	D5405	1	6					Y				
1 *	D5406	1	7					Y				
LCSA WG75857-1	D5407	1	8					Y				
VBLKA	D5408	1	9					N				
VBLKB WG75857-2	D5409	1	10					Y				Target hits
SD1507-13 A	D5410	1	11					Y		<2		
-14 A	D5411	1	12					N				
-1 A	D5412	1	13					N				Gas off
-3 A	D5413	1	14					N				
-5 A	D5414	1	15					N				
-9 A	D5415	1	16					N				
-11 A	D5416	1	17					N				
-15 A	D5417	1	18					N				
-17 A	D5418	1	19					N				
-19 A	D5419	1	20					N				
Rinse	D5420	1	21					—	—	—		
	D5421	1	22					—	—	—		

STANDARD	CODE
BFB	V3048
CAL. STD.	V341
LCS/MS MIX	V342
EXTRAS MIX	V3102

STANDARD	CODE
IS MIX	V3137
SS MIX	V3138

TR 4/5/10

Circle Methods:
SW846 8260
EPA 624
EPA 524
SIM
OLM 04.2
OLM 03.1
OLC 02.1
OLC 03.2

QAMS423
0000072

TITLE: **ANALYSIS OF VOAs BY PURGE AND TRAP GC/MS: SW-846 METHOD 8260**

FIGURE 2

EXAMPLE OF GC/MS STANDARDS RECEIPT LOGBOOK PAGE

KATAHDIN ANALYTICAL SERVICES

STOCK STANDARDS RECEIVED

GCMS LABORATORY
REVIEWED BY/DATE:

AMP1518 1519 1520 1521 — 1-Chlorohexane (EPA-1208) Lot; CB-234, A Ultra Exp; 11/30/09 1000µg/mL — Rec'd 11/07/07

AMP1522 1523 1524 1525 — Restek Cat# 30434 MA VPH Standard 800–1500 ug/ml in Purge and Trap Methanol Lot# A040187 Exp: 11/12 Store: Freezer Restek Corporation - 110 Benner Circle - Bellefonte, PA 16823 — Rec'd 11/7/07 EKL

AMP1526 1527 — Restek Cat# 30465 RL Lot # A049252 Exp: 12/11 Store:Freezer — rec'd DMF 11/20/07

AMP1528 1529 — AccuStandard 125 Market St. - New Haven, CT 06513 - USA Tel: 203-786-5290 - www.accustandard.com AS-E0285 Diethyl ether 5000 µg/mL in MeOH Lot: B4070099-1A Exp. Jan 19, 2010 1 mL FOR LABORATORY USE ONLY STORAGE Ambient POISON — rec'd DMF 11/21/07

AMP1530 1531 — AccuStandard 125 Market St. - New Haven, CT 06513 - USA Tel: 203-786-5290 - www.accustandard.com CLP-LC-IS-100X Laboratory Control Sample - Internal Standard Mix 2500 µg/mL in MeOH Lot: B2090027 Exp: Sep 6, 2012 1 mL 3 comps. FOR LABORATORY USE ONLY STORAGE Ambient POISON

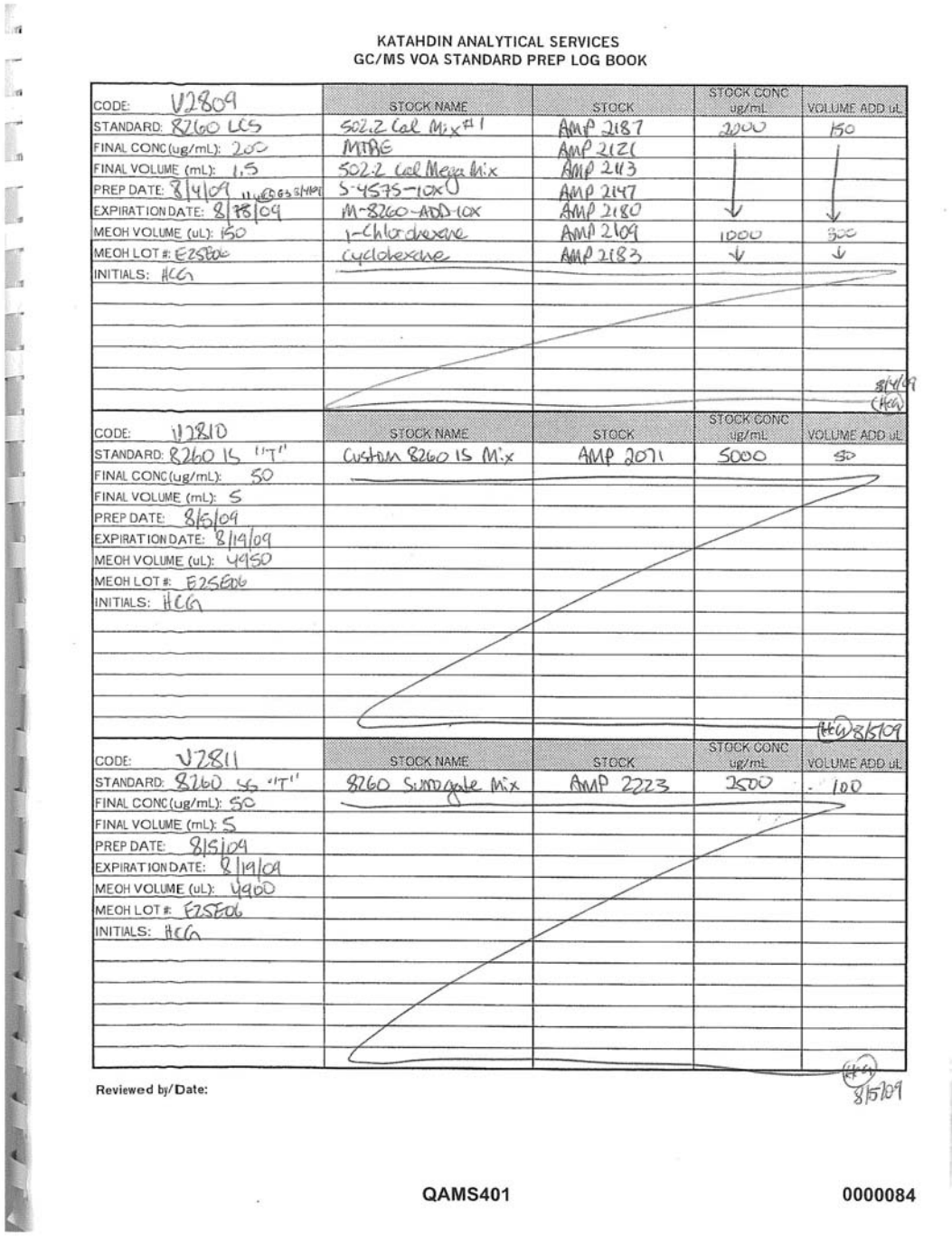
AMP1532 1533 — Restek Cat# 30624 RL Lot # A050631 Exp: 3/10 Store:Freezer — rec'd DMF 11/26/07

QAMS342 0000035

TITLE: **ANALYSIS OF VOAs BY PURGE AND TRAP GC/MS: SW-846 METHOD 8260**

FIGURE 3

EXAMPLE OF VOA STANDARDS PREPARATION LOGBOOK PAGE



KATAHDIN ANALYTICAL SERVICES
GC/MS VOA STANDARD PREP LOG BOOK

CODE: V2809	STOCK NAME	STOCK	STOCK CONC ug/mL	VOLUME ADD uL
STANDARD: 8260 LCS	502.2 Cal Mix #1	AMP 2187	2000	150
FINAL CONC (ug/mL): 20	MTBE	AMP 2121	↓	↓
FINAL VOLUME (mL): 1.5	502.2 Cal Mega Mix	AMP 2143		
PREP DATE: 8/4/09	5-4575-10X	AMP 2147		
EXPIRATION DATE: 8/18/09	M-8260-ADD-10X	AMP 2180	↓	↓
MEOH VOLUME (uL): 150	1-Chlorohexane	AMP 2109	1000	300
MEOH LOT #: E25E06	Cyclohexane	AMP 2183	↓	↓
INITIALS: HCG				
				8/4/09 (HCG)

CODE: V2810	STOCK NAME	STOCK	STOCK CONC ug/mL	VOLUME ADD uL
STANDARD: 8260 IS "T"	Custom 8260 IS Mix	AMP 2071	5000	50
FINAL CONC (ug/mL): 50				
FINAL VOLUME (mL): 5				
PREP DATE: 8/5/09				
EXPIRATION DATE: 8/19/09				
MEOH VOLUME (uL): 4950				
MEOH LOT #: E25E06				
INITIALS: HCG				
				(HCG) 8/5/09

CODE: V2811	STOCK NAME	STOCK	STOCK CONC ug/mL	VOLUME ADD uL
STANDARD: 8260 SG "T"	8260 Surrogate Mix	AMP 2223	2500	100
FINAL CONC (ug/mL): 50				
FINAL VOLUME (mL): 5				
PREP DATE: 8/5/09				
EXPIRATION DATE: 8/19/09				
MEOH VOLUME (uL): 4900				
MEOH LOT #: E25E06				
INITIALS: HCG				
				(HCG) 8/5/09

Reviewed by/Date:

QAMS401 0000084

FIGURE 4

TITLE: **ANALYSIS OF VOAs BY PURGE AND TRAP GC/MS: SW-846 METHOD 8260**

STANDARD INFORMATION

VOA Standards

Standard	Concentration	Manufacturer	Catalog Number
1,2,3 Trimethylbenzene	2000 ug/mL	Restek	58733
1,2,3 Trichlorobenzene	2000 ug/mL	Accustandard	M-502-47-10X
1,2,4 Trimethylbenzene	2000 ug/mL	Accustandard	M-502-54-10X
1,3,5 Trichlorobenzene	neat	Supelco	44-2235
1,3.5 Trimethylbenzene	2000 ug/mL	Accustandard	M502-55-10X
2-CEVE	2000 ug/mL	Accustandard	M-601C-10X
502.2 Cal Mix #1 (gases)	2000 ug/mL	Restek	30042
502.2 Cal2000 Mega Mix	2000 ug/mL	Restek	30431
504.1 Cal Mix	200 ug/mL	Accustandard	M-504.1-CSS
Acrolein & Acrylonitrile	5000 ug/mL	Accustandard	M-603-M-5X
Appendix IX Volatiles Mix	various	Accustandard	M-8240C-R3-10X
Bromochloromethane	2000 ug/mL	Accustandard	M-502-03-10X
California Oxygenates Mix #1	2000 - 10,000 ug/mL	Restek	30465
Carbon Disulfide	2000 ug/mL	Restek	30258
Chloroprene	2000 ug/mL	Accustandard	APPX9-048-R1
Custom GC Std	2000 ug/mL	Accustandard	S-11160
Custom VOC mix	various	Accustandard	S-7920-R1
Custom Volatile GC/MS Std	2000 ug/mL	Accustandard	S-3432B
Custom Volatiles GC/MS	2000 ug/mL	Accustandard	S-3432A
Diethey! Ether	5000 ug/mL	Accustandard	AS E0285
Freon 113	2000 ug/mL	Supelco	4-7944
Method 8260 Additions	2000 ug/mL	Accustandard	M-8260-ADD-10X
Method 8260B-Revision	2000 ug/mL	Accustandard	M-8240B-R-10X
MTBE	2000 ug/mL	Supelco	4-8483
Napthalene	2000 ug/mL	Accustandard	M-502-40-10X
THF	2000 ug/mL	Accustandard	S-4575-10X
Vinyl Acetate	2000 ug/mL	Restek	30216
Vinyl Acetate	2000 ug/mL	Accustandard	APPX9-211-20X
VOA Calibration Mix #1 (Ketones)	5000 ug/mL	Restek	30006
TCL Ketone Mix	5000 ug/mL	Accustandard	CLP-022-25X
VOC Liquid Mix	2000 ug/mL	Accustandard	M-502A-R2-10X
Volatile Organic Compounds (gases)	2000 ug/mL	Accustandard	M-502B-10X
IS/SS/Tune			
Custom 8260 IS	5000 ug/mL	Restek	54577
Custom 8260 SS	5000 ug/mL	Restek	54578
4-BFB	2000 ug/mL	Supelco	48083
VOA Tuning Compound (BFB)	5000 ug/mL	Restek	30003
1,2 Dichlorobenzene-D4	2000 ug/mL	Supelco	48952-U
Fluorobenzene	2000 ug/mL	Supelco	
VOA IS (CLP)	2500 ug/mL	Restek	30004
VOA SS (CLP)	2000 ug/mL	Supelco	48943
624 IS	1500 ug/mL	Restek	30023
4-BFB/Fluorobenzene/Pentafl. (EPA 624)	20000 ug/mL	Accustandard	M-624-SS-M
8260A SS	2500 ug/mL	Restek	30240
CLP Only			
04.1 CLP VOA Cal 2000	2000 ug/mL	Restek	30456
LCS-IS	2500 ug/mL	Accustandard	CLP-LCS-IS-100X
LCS-Volatiles	200 ug/mL	Accustandard	CLP-LCS-V
CLP Volatiles DMC Stock Solution	deutrated compds	Cambridge Isotope	ES 5038
3.2 OLC mix	1000 - 2000 ug/mL	Restek	30492

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY: SW 846 METHOD 8270 – Modified for Selected Ion Monitoring (SIM)

Prepared By: GC/MS Department Date: 6/98

Approved By:

Group Supervisor: J. Halay Date: 020101

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Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
01 8270C Mod.	Format changes, added pollution prevention, added instrument and other calibration options. Other minor changes to sections 7, 8 & QA Table.	DH	1.31.01	1.31.01
02 8270C	Many changes in formatting. Some additions to sections & Table 1 to comply with Navy.	DH	09.30.04	09.30.04
03 8270C	Sect. 7.2: Removed "K" Instrument & added "R" instrument. Added Pentafluorophenol surr. to Tables 3, 5 and Sect. 8.2. Removed all References to TIC's.	LAD	04/06	04/06
04 8270C	Sect. 8.2 - changed 5 to 4 and removed pentachlorophenol. Table 3 and 5 - removed pentachlorophenol. Changed linear regression correlation coefficient criteria. Added MI SOP reference. Added LCS exceedance criteria. Added ICV requirement and criteria. Added RT window procedure.	LAD	06/07	06/07
05 8270C	Added "G" instrument, Removed "X" instrument Edited Section 7.5.1 - initial cal. table	LAD	02/08	02/08

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY: SW 846 METHOD 8270 – Modified for Selected Ion Monitoring (SIM)

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
06	Section 5.3.2.3- Added calibration Mix B. Section 7.5.1- Edited to address diffent SIM compounds may need to be calibrated at different levels depending on the compound and project requirements.	LAD	04/09	04/09
07	Changes made for compliance with DoD QSM version 4.1	LAD	08/09	08/09
08	Updated Standard prep. Added compounds to Table 3 and 5. Updated references. Added DoD QSM QC requirements Table.	LAD	04/10	04/10

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY: SW 846 METHOD 8270 – Modified for Selected Ion Monitoring (SIM)

Please acknowledge receipt of this standard operating procedure by signing and dating both of the spaces provided. Return the bottom half of this sheet to the QA Department.

I acknowledge receipt of copy ___ of document SOP CA-213-08, titled "**ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY METHOD 8270 – Modified for Selected Ion Monitoring (SIM)**".

Recipient: _____ Date: _____

KATAHDIN ANALYTICAL SERVICES, INC.
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy ___ of document SOP CA-213-08, titled "**ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY METHOD 8270 – Modified for Selected Ion Monitoring (SIM)**".

Recipient: _____ Date: _____

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY: SW 846 METHOD 8270 – Modified for Selected Ion Monitoring (SIM)

1.0 SCOPE AND APPLICATION

The purpose of this SOP is to describe the procedures utilized by Katahdin Analytical Services, Inc. laboratory personnel to prepare and analyze water and soil sample extracts for semivolatile organics by EPA SW-846 Method 8270, current revision, modified for selected ion monitoring.

In order to maintain consistency in data quality, this SOP consolidates all aspects of the analyses in one working document, to be revised as necessary.

1.1 Definitions

ANALYTICAL BATCH: 20 or fewer samples which are analyzed together with the same method sequence and the same lots of reagents and with the manipulations common to each sample within the same time period or in continuous sequential time periods.

METHOD BLANK (laboratory reagent blank): An artificial sample designed to determine if method analytes or other interferences are present in the laboratory environment, the reagents, or the apparatus. For aqueous samples, reagent water is used as a blank matrix; for soil samples, baked organic-free sand is used as a blank matrix. The blank is taken through the appropriate steps of the process.

CALIBRATION CHECK: Verification of the ratio of instrument response to analyte amount, a calibration check is done by analyzing for analyte standards in an appropriate solvent. Calibration check solutions are made from a stock solution that is different from the stock used to prepare standards.

CALIBRATION STANDARD (WORKING STANDARD): A solution prepared from the stock standard solution that is used to calibrate the instrument response with respect to analyte concentration.

LABORATORY CONTROL SAMPLE (LCS): A blank that has been spiked with the analyte(s) from an independent source and is analyzed exactly like a sample. Its purpose is to determine whether the methodology is in control, and whether the laboratory is capable of making accurate and precise measurements. The matrix used should be phase matched with the samples and well characterized.

MATRIX SPIKE/MATRIX SPIKE DUPLICATE (MS/MSD): Predetermined quantities of stock solutions of certain analytes are added to a sample matrix prior to sample extraction and analysis. Samples are split into duplicates, spiked and analyzed. Percent recoveries are calculated for each of the analytes detected. The relative percent difference between the samples is calculated and used to assess analytical precision.

STANDARD CURVE (CALIBRATION CURVE): A curve that plots concentration of known analyte standard versus the instrument response to the analyte.

STOCK STANDARD SOLUTION: A concentrated solution containing a single certified standard that is a method analyte, or a concentrated solution of a single analyte prepared in the laboratory with an assay reference compound. Stock standard solutions are used to prepare calibration standards.

SURROGATES: Organic compounds which are similar to analytes of interest in chemical composition, extraction and chromatography, but which are not normally found in environmental samples. These compounds are spiked into all blanks, standards, samples and spiked samples prior to analysis. Percent recoveries are calculated for each surrogate.

TARGET: A software system that combines full processing, reporting and comprehensive review capabilities, regardless of chromatographic vendor and data type.

TARGET DB: An oracle database used to store and organize all Target data files.

QUICKFORMS: A laboratory reporting software for Target and Target DB. The QuickForms report module for Target is preconfigured with generalized forms and US EPA CLP report forms and disk deliverables, which can be customized.

1.2 Responsibilities

This method is restricted to use by, or under the supervision of analysts experienced in the analysis of semivolatile organic compounds by EPA Method 8270. Each analyst must demonstrate and document their ability to generate acceptable results with this method. Refer to Katahdin SOP QA-805, “Personnel Training & Documentation of Capability,” current revision.

It is the responsibility of all Katahdin technical personnel involved in analysis of semivolatiles by Method 8270 to read and understand this SOP, to adhere to the procedures outlined, and to properly document their data in the appropriate lab notebook. Any deviations from the test or irregularities with the samples should also be recorded in the lab notebook and reported to the Department Manager or designated qualified data reviewer responsible for this data.

It is the responsibility of the Department Manager to oversee that members of their group follow this SOP, to ensure that their work is properly documented and to initiate periodic review of the associated logbooks.

1.3 Safety

Users of this procedure must be cognizant of inherent laboratory hazards, proper disposal procedures for contaminated materials and appropriate segregation of hazardous wastes. The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical should be treated as a potential health hazard. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Everyone involved with the procedure must be familiar with the MSDSs for all the materials used in this procedure.

Each qualified analyst or technician must be familiar with the Katahdin Analytical Health and Safety Manual including the Katahdin Hazardous Waste Management Plan and follow appropriate procedures. These include the use of appropriate personal protective equipment (PPE) such as safety glasses, gloves, and lab coats when working with chemicals or near an instrument and not taking food or drink into the laboratory. Each analyst should know the location of all safety equipment. Each analyst shall receive a safety orientation from their Department Manager, or designee, appropriate for the job functions they will perform.

1.4 Pollution Prevention/Waste Disposal

Whenever possible, laboratory personnel should use pollution prevention techniques to address their waste generation. Refer to the current revision of the Katahdin Hazardous Waste Management Program for further details on pollution prevention techniques.

After analysis, autosampler vials containing sample extracts in methylene chloride are returned to the SVOA hood, and the contents transferred to a labeled waste container. The contents of this container are disposed of in accordance with the Katahdin Hazardous Waste Management Plan and Safety Manual and SOP SD-903, “Sample Disposal,” current revision. Expired standards are lab packed, placed in the Katahdin hazardous waste storage area, and disposed of in accordance with this SOP.

2.0 SUMMARY OF METHOD

The process involves the extraction of semivolatiles from a sample using an appropriate solvent followed by clean up steps (where applicable) and concentration of the extract (refer to Katahdin SOP CA-502, “Preparation of Aqueous Samples for Extractable Semivolatile Analyses”, SOP CA-512, “Preparation of Sediment/Soil Samples by Sonication Using Method 3550 for Subsequent Extractable Semi-Volatiles Analysis” and SOP CA-526, “Preparation of Sediment/Soil Samples by Soxhlet Extraction Using Method 3540 for Subsequent Extractable Semivolatile Analysis”). An aliquot of the final extract is injected into the gas chromatograph for compound separation by capillary column, followed by the electron impact mass spectrometer for identification and quantitation.

3.0 INTERFERENCES

Interfering contamination may occur when a sample containing low concentrations of SVOCs is analyzed immediately after a sample containing high concentrations of SVOCs. Any samples that have suspected carryover must be reanalyzed.

4.0 APPARATUS AND MATERIALS

4.1 GC: Hewlett Packard 5890 and/or 6890

4.2 Mass Spectrometers (MS): HP5973, HP5972 and/or HP5970

4.3 Helium: Carrier gas for routine applications. All carrier gas lines must be onstructed from stainless steel or copper tubing; non-polytetrafluoroethylene (non-PTFE) thread sealant or flow controllers with rubber component are not to be used.

4.4 Autosamplers: HP 7673As

4.5 Hamilton syringes: 2.00 uL to 10 mL

4.6 Volumetric glassware: Grade A or equivalent

4.7 Columns: DB-5MS 30m, 0.25mm I.D., 25um film thickness, columns (J&W Scientific) or equivalent.

4.8 Acquisition System: The acquisition system must be interfaced to the MS and allow continuous acquisition of data throughout the duration of the chromatographic program. It must permit, at a minimum, the output of time vs. intensity (peak height or peak area). Hewlett Packard Chemstation or equivalent.

4.9 Data System: The Target software is used for processing data and generating forms.

5.0 REAGENTS

5.1 J.T. Baker Ultra Resi-Analyzed methylene chloride (or equivalent)

5.2 Purge and trap grade methanol

5.3 Standards: Stock standards and working standards are received and recorded in accordance with SOP CA-106 “Standard Preparation and Documentation”.

5.3.1 The expiration date for all standards is one year from date of opening the ampule. If the manufacturer’s expiration date is before this one year date,

the manufacturer's expiration must be followed. New standards must be opened if degradation is observed.

5.3.2 Secondary dilution standards

5.3.2.1 The standards are prepared on an as needed basis (or every 6 months) and stored in screw-cap amber bottles with Teflon liners in the BNA standards freezer between uses. Standards prepared from various stock solutions must always use the first expiration date of any of the solutions used for preparation.

5.3.2.2 Calibration Mix A – Prepare standards in methylene chloride containing the compounds listed in Table 3. The final concentration of each compound is 20 ug/mL.

5.3.2.3 Calibration Mix B - Some compounds must be calibrated at higher concentrations. For these compounds a secondary standard is prepared which will "boost" the concentration of these compounds in the initial calibration. The concentration of this standard is determined on a project to project basis.

5.3.2.4 Internal Standard Solution – Prepare standard in methylene chloride containing 1,4-dichlorobenzene-d4, naphthalene-d8, acenaphthene-d10, phenanthrene-d10, chrysene-d12, and perylene-d12 at a final concentration of 80 ug/mL.

5.3.2.5 DFTPP Solution – Prepare standard in methylene chloride containing DFTPP at a final concentration of 25 ug/mL.

5.3.2.6 Independent Calibration Verification (ICV) Standard – From a source independent of the calibration standards, prepare a standard in methylene chloride containing the compounds listed in Table 3. The final concentration of each compound is 2 ug/mL.

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

All semivolatile sample extracts must be analyzed within forty days following the date of extraction.

7.0 PROCEDURES

7.1 NAMING AND CODING CONVENTIONS FOR ANALYTICAL STANDARDS – Used in accordance with SOP CA-106 "Standard Preparation and Documentation".

7.2 COMPUTER (DATA SYSTEM) CONVENTIONS -

Conventions for all instruments are as follows:

Sub-Directory for data acquisition and storage: C:\HPCHEM\1\DATA
Tune file: DFTPP.U

Method files: LSPSIMXX.M (all samples and standards)
Where:
XX = the calibration number in chronological order
L = instrument ID (R, U, or G)
DFTPP390.M (DFTPP tuning acquisition)

NOTE: All acquisition parameters must be identical for LSPSIMXX.M and DFTPP2. M.

Data Files: L_ _ _ _.D, where _ _ _ _ is a number in chronological order from 0001 to 9999 and L is the instrument ID (R, U, or G). This file also contains the Quantitation output file.

Data Files for DFTPP: LD_ _ _.D, where _ _ _ is a number in chronological order from 001 to 999 and L is the instrument ID (R, U, or G).

7.3 INSTRUMENT SPECIFIC PROCEDURES

It is the policy of the GC/MS group that all data be acquired in the batch mode. The following items must be checked prior to data acquisition in the batch mode:

- Ensure that the proper sequence and tune files are being used.
- Check the autosampler syringe (Is it clean? Does the plunger move freely? etc.), its alignment and make sure the solvent rinse vial is full. Ensure that the knurled nut holding the top of the syringe plunger is tight.
- Look at the batch to be analyzed and check the following:

  -Make sure that the data files are in numerical order with no duplication and that the method file is the same as that used for ICAL or Continuing Calibration analysis.

  -Bottle numbers match with the numbers on the autosampler tray.

After the batch has been deemed free of errors, start the batch by using the “Position and run” command under the SEQUENCE menu in MSTop.

7.4 INSTRUMENT TUNING - Prior to the analysis of any calibration standards, blanks or samples, the GC/MS system must be shown to meet the mass spectral key ion and ion abundance criteria for decafluorotriphenylphosphine (DFTPP) tabulated below. Pentachlorophenol, benzidine and DDT are also present in this standard.

DFTPP Key Ions and Ion Abundance Criteria	
Mass	**Criteria**
51	30.0-80.0 percent of mass 198
68	less than 2.0 percent of mass 69
69	present
70	less than 2.0 percent of mass 69
127	40.0 – 60.0 percent of mass 198
197	less than 1.0 percent of mass 198
198	base peak, 100 percent of mass 198
199	5.0-9.0 percent of mass 198
275	10.0-30.0 percent of mass 198
365	greater than 1.00 percent of mass 198
441	present, but less than mass 443
442	greater than 40.0 percent of mass 198
443	17.0-23.0 percent of mass 442

All ion abundances must be normalized to m/z 198, the nominal base peak.

The following are the GC/MS operating conditions for injection of DFTPP.

GC/MS Operating Conditions - DFTPP	
Initial column temperature hold	140°C for 3 minutes
Column temperature program	140-275°C at 15 degrees/minute
Final column temperature hold	275°C
Injection port temperature	280°C
Transfer line/source temperature	285°C
Injector - splitless, valve time	0.18 minutes
EPC	inlet B
Constant flow	ON
Constant flow pressure	10psi
Constant flow temperature	30°C
Vacuum comp.	ON
Run time	10-12 minutes
Scan start time	5.0 minutes
Sample volume	2.0 uL of 25 ng/uL DFTPP solution
Carrier gas	helium at @ 1.0 mL/minute
Mass range	35 to 500 amu
Number of A/D samples	4
GC Peak threshold	500 counts
Threshold	10 counts

Set up the run on the Enviroquant system using "Edit Sample Log Table". For a more detailed explanation of the Enviroquant software, consult the appropriate manual, Organic Department Manager, or senior chemist within the GC/MS group.

The DFTPP solution must be analyzed once at the beginning of each twelve hour period during which standards and/or samples are analyzed. The 12 hour time period for GC/MS system begins at the moment of injection of the DFTPP analysis. The time period ends after twelve hours has elapsed according to the system clock. The last injection must be accomplished prior to the expiration of 12 hours; conceivably, the run-time of an injection could end after the twelve hours.

When the DFTPP has concluded, the run must be evaluated to determine if sample analysis can proceed. The chromatography and the ion ratios must be examined. The DFTPP run is processed using the current algorithms in the Target software.

If the results indicate the system does not meet acceptance criteria, the GC/MS must be manually tuned. Once the manual tune procedure is completed, DFTPP must be re-injected and reevaluated. If the instrument still does not meet criteria, notify your Department Manager. Under no circumstances should calibration proceed if the instrument DFTPP is not in criteria.

7.5 INSTRUMENT CALIBRATION

7.5.1 Initial Calibration for Method 8270-SIM

Prior to the analysis of samples and required method blanks, and after the instrument DFTPP tuning criteria have been met, the GC/MS system must be calibrated at six different concentrations, typically, 0.20, 0.50, 1.0, 2.0, 5.0 and 8.0 ng/uL. This is done to determine instrument sensitivity and the linearity of GC/MS response for the semivolatile target and surrogate compounds.

Some SIM compounds may need to be calibrated at higher concentrations. A second standard is prepared containing these compounds. The two standards are combined as in the example below. A 100 uL aliquot of each of the standards above is spiked with 1 uL of internal standards and analyzed.

Example –
For a calibration at the following levels:
Calibration mix A would be prepared containing ALL analytes at 20 ng/ul
Calibration Mix B would be prepared containing phenols and phthalates at 20 ng/ul.

Final PAH conc. (ng/uL)	Final Conc. Phenols and phthalates (ng/ul)	Cal-Mix A Added (uL)	Cal-Mix B Added (uL)	$MeCl_2$ Added (uL)	Final Volume (uL)
0.20	1.0	10	40	950	1000
0.50	2.0	25	75	900	1000
1.0	3.0	50	100	850	1000
2.0	4.0	100	100	800	1000
5.0	5.0	250	0	750	1000
8.0	8.0	400	0	600	1000

Note: Calibration Mix B only is used to boost the phenols and phthalates concentrations in Cal. levels 1 through 4.

The GC/MS operating conditions for the calibration standards injections are the same as for the DFTPP with the following exceptions:

GC/MS Operating Conditions – Calibration and Samples	
Column temperature program	40°C for 3 min. to 300°C at 10°/min.
Final column temperature hold	300°C
Run time	35 minutes (time may vary dependent upon column length)
Scan start time	2.0-6.0 minutes (time may vary dependent upon column length)
Sample volume	1 uL

The conditions are set up in the method file LSPSIMXX.M

After analysis of the five calibration points, they must be quantitated and evaluated for adherence to QC criteria. Minimum requirements of ID files are the use of specific quantitation ions and quantitating a specific set of targets and surrogates with a set internal standard. Of particular importance when performing SIM analysis are the ion ratios. These requirements are found in Tables 3 and 5.

7.5.2 Initial Calibration Criteria

Relative response factors (RRFs) must be calculated and evaluated for each target compound and surrogate. The RRF is defined as follows:

$$RRF = \frac{Ax}{A_{IS}} \times \frac{C_{IS}}{Cx}$$

where: A_x = area of the primary ion for the target compound
A_{IS} = area of the primary ion for the corresponding istd
C_{IS} = concentration of the istd (ng/uL)
C_x = concentration of the target compound

After the calibration points have been quantitated, update the calibration curve points using the Target data processing software to generate the RRF's and %RSD's for all analytes. If information is needed concerning the use of these programs, consult the Organic Department Manager or a senior chemist within the group.

Response factor criteria have been established for the calibration of the semivolatile target and surrogate compounds. These criteria must be met in order for the calibration curve to be considered valid. The percent RSD for each calibration check compound (CCC) must be less than or equal to 30 percent. There are three CCC's: Acenaphthene, Fluoranthene, and Benzo(a)pyrene. There are no criteria for the SPCC compounds. This is also applicable to clients that request DOD criteria.

7.5.2.1 Linearity of Target Analytes (This is also applicable to clients that request DOD criteria.)

If the RSD of any target analyte is 15% or less, then the response factor is presumed to be constant over the calibration range, and the average response factor may be used for quantitation.

If the RSD of any target analyte exceeds 15%, then a calibration option outlined in section 7.0 of method 8000 will need to be employed.
Option 1 (Section 7.5.2 of method 8000 - Rev. 2, 12/96), is a linear regression of instrument response versus the standard concentration.

The correlation coefficient (r) for each target analyte and surrogate must be greater than or equal to 0.995. Target software calculates the correlation coefficient squared (r^2). This must be equal to or greater than 0.990.

Option 2 (Section 7.5.3 of method 8000 - Rev. 2, 12/96), is a non-linear calibration model not to exceed a third order polynomial. The lab would use a quadratic model or second order polynomial. The use of a quadratic model requires six calibration points. In order for the quadratic model to be acceptable, the coefficient of determination must be greater than or equal to 0.990.
If time remains in the clock after meeting the initial calibration acceptance criteria, samples may be analyzed. The calibration must be verified each twelve hour time period (time period starts from the

moment of the DFTPP injection) for Method 8270-SIM. The SSTD1.0 in the curve may be used as the continuing calibration standard as long as it meets the continuing calibration acceptance criteria. All sample results must be quantitated using the initial calibration response factors.

7.5.2.2 Immediately following calibration an Independent Calibration Verification Standard must be analyzed. For clients requiring DOD criteria, all project analytes must be within +/- 20% of true value.

7.5.3 Continuing Calibration

A check of the calibration curve must be performed once every twelve hours immediately following analysis of the tuning compound DFTPP. This check contains all target compounds and surrogates at a concentration of 1.0 ng/uL.

After quantitation of the 1.0 ng/uL continuing calibration check, response factors must be calculated and compared to the average response factors in the initial calibration. The Target program calculates the calibration check response factors and compares them to the average RFs in the calibration curve by calculating percent differences. The method 8270 CCC's must have a % difference of +/- 20%D in order to be considered in criteria. These conditions must be met before method blank and/or sample analysis can begin. For clients requiring DOD criteria, all project analytes and surrogates must be within +/- 20%.

If the continuing calibration check does not meet criteria, corrective action must be taken. Depending on the situation, corrective action may be as follows:

- Re-analyze the 1.0 ng/uL continuing calibration check.
- Change the septum; clean the injection port; install a clean, silanized quartz liner; cut off a small portion (1" to 3") of the front end of the capillary column. This is usually performed when chromatography is poor. Record any of these actions in the appropriate instrument maintenance logbook.

- Analyze a new initial calibration curve.

The last option, the generation of a new initial calibration curve, is usually chosen when percent difference are >30%. In these instances, there is little or no chance of a continuing calibration reanalysis meeting criteria. If there is any doubt concerning which corrective action to undertake, consult the Organic Department Manager or a senior chemist within the group.

If the continuing calibration does meet the criteria specified above then analysis may proceed using initial calibration response factors.

7.5.4. Retention Time Windows

Retention time windows are set at the midpoint standard of the calibration curve, following every ICAL. When a CV is analyzed (and not an ICAL), the retention time windows of the daily CV must be within 30 seconds of the midpoint calibration standard of the most recent ICAL. The samples analyzed following the daily CV must have retention times within 30 seconds of those for the daily CV. Each successive daily CV must be compared to the most recent ICAL midpoint standard.

7.6 SAMPLE ANALYSIS

Sample extracts may be analyzed only after the GC/MS system has met tuning criteria, initial calibration and continuing calibration requirements. Ensure that the same instrument conditions are being used for tuning, calibration and sample analysis by reviewing the GC parameters using the "Edit entire method" option under the Method menu in MSTOP. Note that you can not edit a method if the instrument is running.

Extracts are stored in the refrigerator in the organics extraction laboratory at 4°C ±2°C. Remove them from the refrigerator and place them in the GC/MS laboratory semivolatile hood when ready for analysis.

Prepare a 1.8 mL clear glass vial (crimp top) with a disposable insert (350 uL). Add 100 uL of sample extract and 1.0 uL of the 80 ng/uL IS stock to the vial and then cap. This gives a 0.8 ng/uL final concentration for the internal standard compounds. The samples are topped with Teflon lined crimp top caps.

7.7 FINAL DATA PACKAGE

7.7.1 Initial Data Review (IDR)

The initial data review is accomplished by the analyst who ran the samples and is a review of sufficient quality and detail to provide a list of samples that need to be reanalyzed or diluted and reanalyzed. The initial data review is performed on the detailed quantitation reports of the analyzed sample. This data review examines criteria that directly impact whether or not the sample needs to be reanalyzed:

- Surrogate Recoveries
- Internal Standard Area Stability

- Method Blank Acceptance
- Chromatography
- Target Compound Detection/Quantitation/Review for false positives
- Laboratory Control Sample Recoveries
- Matrix Spike/Matrix Spike Duplicate Recoveries

The analyst must evaluate all data using the QA Acceptance Criteria table found within this SOP (Table 1). This table gives acceptance criteria and corrective actions for criteria that are not met. In addition to evaluating QC elements, the chromatography and quantitation of target analytes must be reviewed. During this review, the analyst checks the integration of each individual peak. The hardcopy has false positives crossed out so they can be reviewed for appropriateness by the Organic Department Manager.

7.7.2 Chromatography

The chromatography should be examined for the presence or absence of any ghost peaks and can also be used as an indication of whether or not matrix interferences might be affecting surrogate recoveries and/or istd area recoveries. Whether or not the chromatography is acceptable is a judgment call on the part of the analyst and should be used in conjunction with other monitored QC (e.g. surrogate recoveries) to determine the necessity of reanalyzing.

Manual integrations are to be performed when chromatographic conditions preclude the computer algorithm from correctly integrating the peak of concern. In no instance shall a manual integration be performed solely to bring a peak within criteria.

Each peak of concern is examined by the primary analyst to ensure that the peak was integrated properly by the computer algorithm. Should a manual integration be necessary (for instance, due to a split peak, peak tailing, or incomplete resolution of isomeric pairs), an “m” qualifier will automatically be printed on the quantitation report summary.

This manual integration package must then be submitted to the Department Manager or his/her designee, who will review each manual integration.

For specific manual integration procedures, refer to Katahdin SOP QA-812, “Manual Integration”, current revision.

7.7.3 Target Compound Detection/Quantitation

The semivolatile ID files have been set up to err on the side of false positives; that is to identify and quantitate peaks as target compounds that may not necessarily be valid hits. It is the responsibility of the GC/MS analyst to use his/her technical judgment to determine if the identification of a target compound is correct or not.

If any target concentration exceeds the upper limit, a dilution must be made and analyzed. The dilution chosen should keep the concentration of the largest target compound hit in the upper half of the initial calibration range. LCS and MS/MSD samples need not be diluted to get spiked analytes within the calibration range.

The requirements for qualitative verification by comparison of mass spectra are as follows:

- All ions present in the standard mass spectra at a relative intensity > 10% must be present in the sample spectrum.
- The relative intensities of primary and secondary ions must agree within ±20% between the standard and sample spectra.
- Ions greater than 10% in the sample spectrum but not present in the standard spectrum must be considered and accounted for by the analyst.

If a compound cannot be verified by all three criteria above, but, in the technical judgment of the mass spectral interpretation specialist, the identification is correct, then the laboratory shall report that compound on the Form 1 as a valid hit.

The GC/MS laboratory initial data review must be completed within twelve hours of batch completion; in the majority of instances, the initial review should be accomplished at the beginning of a work shift for the previous set of analyses.

7.7.4 Reporting

After the chromatograms have been reviewed and any target analytes have been quantitated using Target, the necessary files are brought into QuickForms. Depending on the QC label requested by the client, a Report of Analysis (ROA) and additional reports, such as LCS forms and chronology forms, are generated. The package is assembled to include the necessary forms and raw data. The data package is reviewed by the primary analyst and then forwarded to the secondary reviewer. The secondary reviewer validates the data and checks the package for any errors. When completed, the package is sent to the department manager for final review. A complete review checklist is provided with each package. The final data package from

the Organics department is then processed by the Data Management department.

7.8 INJECTION PORT LINER CLEANING AND SILANIZING PROCEDURE

- Remove the rubber o-ring from the liner and place the liner in a large Erlenmeyer flask.
- In the hood, pour nitric acid into the flask until the liner is covered. Place the flask on a hotplate and boil for 2-3 hours.
- Let cool; drain nitric acid and thoroughly flush the liner with water.
- Bake briefly in the muffle oven until liner is dry and cool to room temperature.
- Place the liner in a beaker, fill with Sylon and let it soak for at least two hours.
- Take out the liner and rinse it thoroughly with toluene.
- Rinse the liner thoroughly with purge and trap grade methanol.
- Bake the liner in the muffle oven for a minimum of three hours.

7.9 Instrument Maintenance

Instrument preventative maintenance is performed on a semi-annual basis by GC/MS chemists. This maintenance includes a thorough inspection and cleaning of all parts, including changing rough and turbopump oils. GC/MS analysts perform other maintenance on an as-needed basis. Typically, routine maintenance involves clipping off the front end of the DB-5MS column, replacing the injection port septum, and installing a freshly silanized quartz liner after sample analysis.

All maintenance must be documented in the instrument-specific maintenance log, whether it is routine or not. The Department Manager must authorize any maintenance over and above a routine source cleaning.

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

Refer to Table 1 and to details in this section for a summary of QC requirements, acceptance criteria, and corrective actions. These criteria are intended to be guidelines for analysts. The criteria does not cover all possible situations. If any of the QC requirements are outside the recovery ranges listed in this section or in Table 1, all associated samples must be evaluated against all the QC. In some cases data may be reported, but may be reanalyzed in other cases. Making new reagents and standards may be necessary if the standardization is suspect. The corrective actions listed in this section and in Table 1 may rely on analyst experience to make sound scientific judgements. These decisions are based on holding time considerations, client and project specific Data Quality Objectives and on review of chromatograms. The supervisor, Operations Manager, and/or Quality Assurance Officer may be consulted to evaluate data. Some samples may not be able to be reanalyzed within hold time. In these cases “qualified” data with narration may be advisable after consultation with the client.

In some cases the standard QC requirements listed in this section and in Table 1 may not be sufficient to meet the Data Quality Objectives of the specific project. Much of the work performed at the lab is analyzed in accordance with specific QC requirements spelled out in a project specific Quality Assurance Project Plan (QAPP) or in a program specific Quality Systems Manual (QSM). The reporting limits, acceptance criteria and/or corrective actions may be different than those specified in this SOP. In these cases the appropriate information will be communicated to the Department Manager and/or senior chemists before initiation of the analyses so that specific product codes can be produced for the project. In addition, the work order notes for each project will describe the specific QAPP or QSM to be followed.

8.1 Method Blank Criteria

A method blank is defined as a volume of a clean reference material (deionized distilled water for water samples, baked organic-free sand for soil/sediment matrices) that is carried through the entire analytical procedure. One method blank must be extracted with each group of samples of a similar matrix and must be analyzed on the GC/MS system that was used to analyze the samples.
An acceptable method blank must contain less than or equal to the PQL of any target compound. For clients requiring DOD criteria, no analytes detected at > ½ PQL and > 1/10 the amount measrued in any sample or 1/10 the regulatory limit.

If the method blank exceeds these contamination levels, the analytical system is considered out of control and corrective action must be taken before sample analysis.

Reanalysis of the blank is the first step of the corrective action; if that does not solve the problem, a Katahdin Corrective Action Report (CAR) will be initiated. Corrective action will be specified after consultation including the Department Manager, Operations Manager, and QA Officer.

8.2 Surrogate Recoveries

The four surrogates (2-Methylnaphthalene-d10, 2,4-Dibromophenol, Fluorene-d10 and Pyrene-d10) must meet the current statistically derived acceptance limits. If statistical limits have not been established then the surrogate recovery must meet the nominal limits of 30-150%. For clients requiring DOD criteria, use acceptance limits specified by DOD or use in-house limits where none are specified.

If specifications are not met, the sample (or blank) should be reanalyzed. If specifications are met in the reanalysis, this reanalysis should only be submitted. If surrogate specifications are not met in the sample or method blank reanalysis, a Corrective Action Report (CAR) should be initiated. Corrective action will be specified after consultation including the Department Manager and Operations Manager.

For further information regarding the acceptance of surrogate recoveries, consult the Organic Department Manager.

8.3 Internal Standard Responses

Internal standard responses and retention times (RT) in all samples and blanks must be evaluated as part of the technical data review. The method files have been set up to only detect compounds that fall within a set RT window. For Method 8270-SIM analysis, if the extracted ion current profile (EICP) area for any internal standard changes by more than a factor of two (-50% to +100%) as compared to the daily continuing calibration standard, reanalysis must occur. If the reanalysis meets criteria, only the in-criteria run should be reported. If the reanalysis is still out of criteria, both analyses should be included in the sample package set.
MS/MSD samples that do not meet the EICP area criteria above do not have to be reanalyzed.

8.4 Laboratory Control Sample (LCS)

An LCS must be performed for each group of samples of a similar matrix, for the following, whichever is more frequent:

- Every 20 samples of a similar matrix or similar concentration, or
- Every batch of samples extracted.

Statistical limits are compiled annually for LCS recoveries (archived in QA office). Statistical limits are only calculated when at least 20 usable data points are obtained for any given compound. If insufficient data points are available, nominal limits are set by the section supervisor, Laboratory Operations Manager and Quality Assurance Officer. Refer to Katahdin SOP QA-808, “Generation and Implementation of Statistical QC Limits and/or Control Charts”, current revision.

The use of statistical limits versus nominal limits is dependent on the client and project. This information is communicated to the Organic Department Manager through the Katahdin project manager. It is standard practice to use statistical limits for reporting purposes and to evaluate any QC criteria exceedances. However, nominal limits of 30-150% may be used for some projects or states (i.e. South Carolina). For clients requiring DOD criteria, use acceptance limits specified by DOD or use in-house limits where none are specified.

The LCS recoveries for all analytes are evaluated. All of the compounds of interest must fall within the established statistical limits with the following sporadic exceedance allowances.

# of Analytes	# of Allowable Exceedances
> 90	5
71 – 90	4
51 – 70	3
31 – 50	2

# of Analytes	# of Allowable Exceedances
11 – 30	1
<11	0

If less than the number of allowable exceedances fail the statistical limits, no corrective action is needed. If greater than the number of allowable exceedances fail the statistical limits, corrective action may be taken. Corrective actions may vary with each situation. However, in the case where the failures are high and the samples are non-detect for those compounds, then no corrective action is required. Otherwise, corrective action may involve reanalysis or recalibration. The specific corrective actions taken will rely on analyst experience to make sound scientific judgments while considering client objectives, other quality control indicators and/or the ability to reanalyze a sample within holding time

Please note that for compounds with only nominal limits (i.e. insufficient data points were available to generate statistical limits), no corrective action is required for out-of-criteria recoveries until enough data points are established to generate statistical limits.

8.5 Matrix Spike/Matrix Spike Duplicate (MS/MSD) Criteria

Matrix Spike and Matrix Spike Duplicates must be extracted and analyzed for each group of up to 20 samples of a similar matrix or similar concentration. In the event insufficient sample volume is available an LCS/LCS Duplicate is extracted and analyzed in place of the MS/MSD.

Statistical limits are compiled annually for MS/MSD recoveries for a short list of the spiked compounds (Acenaphthene, Pentachlorophenol and Pyrene). Nominal limits of 30-130% are used for all other compounds. Generally, corrective action is only taken for the short list of the spiked compounds. The specific corrective actions will rely on analyst experience to make sound scientific judgments while considering client objectives, other quality control indicators and/or the ability to reanalyze a sample within holding time. For clients requiring DOD criteria, use acceptance limits specified by DOD or use in-house limits where none are specified.

A Corrective Action Report (CAR) must be filled out and filed if any criteria for percent recovery or relative percent difference are not met to document any decisions with reporting data.

9.0 METHOD PERFORMANCE

The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero. The MDLs are determined annually per type of instrument and filed with the Organic Department

Manager and with the QAO. Refer to the current revision of Katahdin SOP QA-806, Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications, for procedures on determining the MDL.

Refer to the current revision of Method 8270 for other method performance parameters and requirements.

10.0 APPLICABLE DOCUMENTS/REFERENCES

"Test Methods for the Evaluation of Solid Waste: Physical/Chemical Methods", SW-846, third Edition, Final Updates I, II, IIA, III, IIIA and IIIB, November 2004, Method 8270C.

Department of Defense Quality Systems Manual for Environmental Laboratories (DoD QSM), Version 4.1, 04/22/09

The National Environmental Laboratory Accreditation Conference (NELAC) Standards, June 2003

"USEPA Contract Laboratory Program Statement of Work for Organics Analysis," Rev. 02/88.

Code of Federal Regulations (40 CFR), Part 136, Appendix A, Rev. June, 1998.

Katahdin SOP CA-101, Equipment Maintenance and Troubleshooting, current revision.

Katahdin SOP QA-806, Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications, current revision.

LIST OF TABLES AND FIGURES

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY: SW 846 METHOD 8270 – Modified for Selected Ion Monitoring (SIM)

TABLE 1

QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Check of mass spectral ion intensities using DFTPP	Prior to initial calibration and calibration verification	Refer to the criteria listed in Section 7.4	Retune instrument, and verify
Six-point initial calibration for all analytes	Initial calibration prior to sample analysis	RSD ≤30 for RFs of the CCCs; Average %RSD < 15% for all compounds. Refer to section 7.5.2.1 for more details.	Repeat calibration if criterion is not met
Independent calibration verification	Once after Initial calibration	± 20 % D	1) Reanalyze standard 2) Reprep standard 3) Reprep standard from fresh stock.
Continuing calibration verification	Once per each 12 hours, prior to sample analysis	CCCs ≤ 20%D	Repeat initial calibration and reanalyze all samples analyzed since the last successful calibration verification
ISs	Immediately after or during data acquisition of calibration check standard	Retention time ± 30 seconds; EICP area within -50% to +100% of last calibration verification (12 hours) for each IS	Inspect mass spectrometer or GC for malfunctions; mandatory reanalysis of samples analyzed while system was malfunctioning
Demonstration of ability to generate acceptable accuracy and precision	Once per analyst initially and annually thereafter	All recoveries within method QC acceptance limits.	Recalculate results; locate and fix problem; reextract/reanalyze P&A study for those analytes that did not meet criteria
Method blank	One per prep batch	No analytes detected > PQL	(1) Investigate source of contamination (2) Evaluate the samples and associated QC: i.e.If the blank results are above the PQL, report samples that are <PQL or > 10X the blank result. Reprep a blank and the remaining samples.
LCS for all analytes	One LCS per prep batch	Statistically derived from lab data or nominal limits depending on the project. . See also section 8.4 of this SOP for more information on allowable exceedances.	(1) Evaluate the samples and associated QC: i.e.If an MS/MSD was performed and acceptable, narrate. If an LCS/LCSD was performed and only one was unacceptable, narrate. If the surrogate recoveries in the LCS are low but are acceptable in the blank and samples, narrate. If the LCS rec. is high but the sample results are <PQL, narrate. Otherwise, reprep a blank and the remaining samples.

TABLE 1

QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Surrogate spike	Every sample, control, standard, and method blank	Statistically derived limits.	(1) Check chromatogram for interference; if found, flag data (2) If not found, check instrument performance; if problem is found, correct and reanalyze (3) If still out reextract and analyze sample (4) If reanalysis is out, flag data
MS/MSD	One MS/MSD per every 20 samples	Statistically derived from lab data or nominal limits depending on the project. Nominal limits are used as default limits.	(1) Evaluate the samples and associated QC: i.e. If the LCS results are acceptable, narrate. (2) If both the LCS and MS/MSD are unacceptable reprep the samples and QC.
MDL and/or LOD/LOQ Verification study	Refer to KAS SOP QA-806, "Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications", current revision.		

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY: SW 846 METHOD 8270 – Modified for Selected Ion Monitoring (SIM)

TABLE 2

DoD QSM QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Demonstrate acceptable analytical capability	Prior to using any test method and at any time there is a significant change in instrument type, personnel, test method, or sample matrix.	QC acceptance criteria published by DoD, if available; otherwise, method-specific criteria.	Recalculate results; locate and fix problem, then rerun demonstration for those analytes that did not meet criteria.	NA.	This is a demonstration of analytical ability to generate acceptable precision and bias per the procedure in Appendix C. No analysis shall be allowed by analyst until successful demonstration of capability is complete.
LOD determination and verification	Refer to current revision of SOP QA-806				
LOQ establishment and verification	Refer to current revision of SOP QA-806				
Tuning	Prior to ICAL and at the beginning of each 12-hour period.	Refer to method for specific ion criteria.	Retune instrument and verify. Rerun affected samples.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be accepted without a valid tune.
Breakdown check (DDT Method 8270 only)	Correct problem then repeat breakdown check.	Degradation ≤ 20% for DDT. Benzidine and pentachlorophenol should be present at their normal responses, and should not exceed a tailing factor of 2.	At the beginning of each 12-hour period, prior to analysis of samples.	Flagging criteria are not appropriate.	No samples shall be run until degradation ≤ 20%.
Minimum five-point initial calibration (ICAL) for all analytes	ICAL prior to sample analysis.	1. Average response factor (RF) for SPCCs ≥ 0.050. 2. RSD for RFs for CCCs ≤ 30% and one option below: Option 1: RSD for each analyte ≤ 15%; Option 2: linear least squares regression r ≥ 0.995; Option 3: non-linear regression– coefficient of determination (COD) r2 ≥ 0.99 (6 points shall be used for second order).	Correct problem then repeat ICAL.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be run until ICAL has passed. Calibration may not be forced through the origin.
Second source calibration verification (ICV)	Once after each ICAL.	All project analytes within ± 20% of true value.	Correct problem and verify second source standard. Rerun second source verification. If that fails, correct problem and repeat ICAL.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be run until calibration has been verified.

TABLE 2 (cont)

DoD QSM QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Retention time window position establishment for each analyte and surrogate	Once per ICAL.	Position shall be set using the midpoint standard of the ICAL curve when ICAL is performed. On days when ICAL is not performed, the initial CCV is used.	NA.	NA.	
Evaluation of relative retention times (RRT)	With each sample.	RRT of each target analyte within ± 0.06 RRT units.	Correct problem, then rerun ICAL.	Flagging criteria are not appropriate.	Laboratories may update the retention times based on the CCV to account for minor performance fluctuations or after routine system maintenance (such as column clipping). With each sample, the RRT shall be compared with the most recently updated RRT. If the RRT has changed by more than ±0.06 RRT units since the last update, this indicates a significant change in system performance and the laboratory must take appropriate corrective actions as required by the method and rerun the ICAL to reestablish the retention times.
Continuing calibration verification (CCV)	Daily before sample analysis and every 12 hours of analysis time.	1. Average RF for SPCCs ≥ 0.050. 2. %Difference/Drift for all target compounds and surrogates ≤ 20%D (Note: D = difference when using RFs or drift when using least squares regression or non-linear calibration).	DoD project level approval must be obtained for each of the failed analytes or corrective action must be taken. Correct problem, then rerun calibration verification. If that fails, then repeat ICAL. Reanalyze all samples since last acceptable CCV.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to all results for the specific analyte(s) in all samples since last acceptable CCV.	Problem must be corrected. Results may not be reported without a valid CCV. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Internal standards verification	Every field sample, standard, and QC sample.	Retention time ± 30 seconds from retention time of the midpoint standard in the ICAL; EICP area within -50% to +100% of ICAL midpoint standard.	Inspect mass spectrometer and GC for malfunctions. Reanalysis of samples analyzed while system was malfunctioning is mandatory.	If corrective action fails in field samples, apply Q-flag to analytes associated with the non-compliant IS. Flagging criteria are not appropriate for failed standards.	Sample results are not acceptable without a valid IS verification.

TABLE 2 (cont)

DoD QSM QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Method blank	One per preparatory batch.	No analytes detected > ½ RL (> RL for common lab contaminants) and > 1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater). Blank result must not otherwise affect sample results.	Correct the problem. Report sample results that are <LOD or >10x the blank concentration. Reprepare and reanalyze the method blank and all associated samples with results > LOD and < 10x the contaminated blank result. Contact Client if samples cannot be reprepped within hold time.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply B-flag to all results for the specific analyte(s) in all samples in the associated preparatory batch.	Problem must be corrected. Results may not be reported without a valid method blank. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
LCS containing all analytes to be reported, including surrogates	One per preparatory batch.	The laboratory shall use laboratory control limits (CLs) or use DoD-generated LCS-CLs, if available depending on project requirements. In-house CLs may not be greater than ± 3 times the standard deviation of the mean LCS recovery. A number of analytes may fall outside the CL but within marginal exceedance limit depending on the total number of analytes in the LCS.	Correct problem, then reprep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available. Refer to Table G-1 for number of marginal exceedences allowed. Contact Client if samples cannot be reprepped within hold time.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch.	Problem must be corrected. Results may not be reported without a valid LCS. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Matrix Spike (MS)	One per preparatory batch per matrix if sufficient sample is available.	For matrix evaluation, use LCS acceptance criteria.	Examine the project-specific DQOs. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met.	For matrix evaluation only. If MS results are outside the LCS limits, the data shall be evaluated to determine the source of difference and to determine if there is a matrix effect or analytical error.
Matrix spike duplicate (MSD) or sample duplicate	One per preparatory batch per matrix if sufficient sample is available.	MSD: For matrix evaluation, use LCS acceptance criteria. MS/MSD: RPD ≤ 30%.	Examine the project-specific DQOs. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met.	The data shall be evaluated to determine the source of difference.

TABLE 2 (cont)

DoD QSM QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Surrogate spike	All field and QC samples.	The laboratory shall use laboratory surrogate CLs or use DoD-generated surrogate CLs, if available depending on project requirements. .	For QC and field samples, correct problem then reprep and reanalyze all failed samples for failed surrogates in the associated preparatory batch, if sufficient sample material is available. If obvious chromatographic interference with surrogate is present, reanalysis may not be necessary. Contact Client if samples cannot be reprepped within hold time.	Apply Q-flag to all associated analytes if acceptance criteria are not met.	Alternative surrogates are recommended when there is obvious chromatographic interference.
Results reported between DL and LOQ	NA.	NA.	NA.	Apply J-flag to all results between DL and LOQ.	

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY: SW 846 METHOD 8270 – Modified for Selected Ion Monitoring (SIM)

TABLE 3

SUMMARY OF METHOD MODIFICATIONS

TOPIC	KATAHDIN SOP CA-213-08	METHOD 8270, current revision
Apparatus/Materials	none	
Reagents	none	
Sample preservation/ handling	none	
Procedures	none	
QC - Spikes	none	
QC - LCS	none	
QC - Accuracy/Precision	none	
QC - MDL	none	

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY: SW 846 METHOD 8270 – Modified for Selected Ion Monitoring (SIM)

TABLE 4

ANALYTE QUANITIATION AND INTERNAL STANDARDS

Internal Standard: 1,4-dichlorobenzene-d4
Target and Surrogates:
1,4-Dioxane
Benzaldehyde
Phenol
bis(2-Chloroethyl)ether
2-Chlorophenol
2-Methylphenol
3&4-Methylphenol
2,2'-Oxybis(1-chloropropane)
Nitrobenzene
Hexachloroethane
Acetophenone
N-nitroso-di-n-propylamine
Internal Standard: Naphthalene-d8
Target and Surrogates:
Naphthalene
1-Methylnaphthalene (dredge)
2-Methylnaphthalene
2-Methylnaphthalene-D10 (surrogate)
Isophorone
2-Nitrophenol
2,4-Dimethylphenol
bis(2-Chloroethoxy)methane
2,4-Dichlorophenol
4-Chloroaniline
Hexachlorobutadiene
Caprolactam
4-Chloro-3-methylphenol
Internal Standard: Acenaphthene-d10
Target and Surrogates:
1,1'-Biphenyl (dredge)
2,6 Dimethylnapthalene (dredge)
Acenaphthylene
Acenaphthene
Fluorene
2-Fluorene-d10 (surrogate)
2,4-Dibromophenol (surrogate)
2-Chloronaphthalene
Hexachlorocyclopentadiene
2,4,6-Trichlorophenol
2,4,5-Trichlorophenol
Dimethylphthalate
2,6-Dinitrotoluene

2,4-Dinitrotoluene
2,4-Dinitrophenol
2,3,4,6-Tetrachlorophenol
Diethylphthalate
4-Chlorophenyl-phenyl ether
4,6-Dinitro-2-methylphenol
N-nitrosodiphenylamine
2-Nitroaniline
3-Nitroaniline
4-Nitroaniline
Dibenzofuran
4-Nitrophenol
Internal Standard: Phenanthrene-d10
Target and Surrogates:
Pentachlorophenol
1-Methylphenanthrene (dredge)
Phenanthrene
Hexachlorobenzene (special)
Anthracene
Fluoranthene
Carbazole
Di-n-butylphthalate
4-Bromophenyl-phenyl ether
Atrazine
Internal Standard: Chrysene-d12
Target and Surrogates:
Butylbenzylphthalate
3,3'-Dichlorobenzidine
Pyrene
Benzo(a)Anthracene
Chrysene
Bis-(2-ethylhexyl)phthalate
Pyrene-d10 (surrogate)
Internal Standard: Perylene-d12
Target and Surrogates:
Perylene (dredge)
Benzo(b)fluoranthene
Benzo(k)fluoranthene
Benzo(e)pyrene (dredge)
Di-n-octylphthalate
Benzo(a)pyrene
Indeno(1,2,3-cd)pyrene
Dibenz(a,h)anthracene
Benzo(ghi)perylene

TABLE 5

PROCEDURE CONDENSATION

Clock

12 hours from injection of 50ng DFTPP.

Calibration Curve Criteria

<30% RSD for CCCS
<15% RSD average for all analytes in calibration standard

Continuing Calibration Check Criteria

<20% D for CCC compounds

Additional QC

LCS every extraction batch
MS/MSD every 20 samples

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY: SW 846 METHOD 8270 – Modified for Selected Ion Monitoring (SIM)

TABLE 6

SVOA COMPOUNDS AND CHARACERISTIC IONS

COMPOUND	PRIMARY ION	SECONDARY IONS
1,4-Dioxane	88	58
Benzaldehyde	77	105,106
Phenol	94	65,66
bis(2-Chloroethyl)ether	93	63,95
2-Chlorophenol	128	64,130
1,4-Dichlorobenzene-d4 (IS)	152	150,115
2,2'-Oxybis(1-choropropane)	45	77,121
2-Methylphenol	108	107,77
Acetophenone	105	77,51
N-nitroso-di-n-propylamine	70	52,101
Hexachloroethane	117	201,199
3&4-Methylphenol	108	107,77
Nitrobenzene	77	123,51
Isophorone	82	54,138
2-Nitrophenol	139	109,81
2,4-Dimethylphenol	107	122,121
bis(2-Chloroethoxy)methane	93	63,123
2,4-Dichlorophenol	162	164,98
Naphthalene-d8 (IS)	136	137,134
Naphthalene	128	129,127
4-Chloroaniline	127	129
Hexachlorobutadiene	225	223,227
Caprolactam	113	55,56
4-Chloro-3-methylphenol	107	77,142
2,4-Dibromophenol (surr)	252	63,143
2-Methylnaphthalene-d10 (surr)	152	150
2-Methylnaphthalene	142	141,115
1-Methylnaphthalene	142	141,115
Hexachlorocyclopentadiene	237	235,239
2,4,6-Trichlorophenol	196	198,200
2,4,5-Trichlorophenol	196	198,200
2-Chloronaphthalene	162	127,164
1,1'-Biphenyl	154	153,76
2-Nitroaniline	65	92,138
Dimethylphthalate	163	194,164
2,6-Dinitrotoluene	165	63,89
Acenaphthylene	152	151,153
Acenaphthene	152	154,152
Acenaphthene-d10 (IS)	164	162
3-Nitroaniline	138	65,92
2,4-Dinitrophenol	184	107
Dibenzofuran	168	139

TABLE 6 (cont.)

SVOA COMPOUNDS AND CHARACERISTIC IONS

COMPOUND	PRIMARY ION	SECONDARY IONS
2,4-Dinitrotoluene	165	63
4-Nitrophenol	109	139,65
2,3,4,6-Ttrachlorophenol	232	230
Diethylphthalate	149	177,176
Fluorene-d10 (surr)	176	174,178
Fluorene	166	165
4-Chlorophenyl-phenyl ether	204	206,141
4-Nitroaniline	138	108,65
4,6-Dinitro-2-methylphenol	198	121
N-nitrosodiphenylamine	169	168,167
4-Bromophenyl-phenyl ether	248	250,141
Hexachlorobenzene	284	142,249
Atrazine	200	173,215
Pentachlorophenol	266	264,268
Phenanthrene-d10 (IS)	188	189
Phenanthrene	178	179,176
Anthracene	178	179,176
Carbazole	167	166,139
Di-n-butylphthalate	149	150,104
Fluoranthene	202	200,203
Pyrene	202	200,201
Pyrene-d10 (surr)	212	210,106
Butylbenzylphthalate	149	91,206
Benzo(a)anthracene	228	229,226
Chrysene-d12 (IS)	240	236,120
3,3-Dichlorobenzidine	252	254,126
Chrysene	228	226,229
bis(2-Ethylhexyl)phthalate	149	167
Di-n-octylphthalate	149	150
Benzo(b)fluoranthene	252	253,125
Benzo(k)fluoranthene	252	253,125
Benzo(a)pyrene	252	253,250
Perylene-d12 (IS)	264	260
Indeno(1,2,3-cd)pyrene	276	277
Dibenzo(a,h)anthracene	278	279
Benzo(g,h,i)perylene	276	277

Primary ions must not be changed except in unusual instances where interference occurs with a co-eluting non-target analyte. In this case, a secondary ion may be used for quantitation with the following rules:
(1) The corresponding standard(s) (initial calibration curve and continuing calibration standard) must be re-quantitated with the secondary ion.

TABLE 6 (cont.)

SVOA COMPOUNDS AND CHARACERISTIC IONS

(2) Approval must be obtained from the Organic Department Manager or the laboratory Operations Manager.

The quantitation ion must then be changed back to the one specified in the table above after quantitation of the samples(s).

Secondary ions are recommended only and may be changed depending upon instrument conditions (sensitivity, etc.). However, it is Katahdin policy that a minimum of 2 ions (primary and one secondary) be used for all GC/MS analyses.

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY: SW 846 METHOD 8270 – Modified for Selected Ion Monitoring (SIM)

FIGURE 1

EXAMPLE OF RUNLOG LOGBOOK PAGE

KATAHDIN ANALYTICAL SERVICES
GC/MS SVOA INJ LOG INSTRUMENT: 5970-X

DATE OF DFTPP INJECTION: 03/06/09

JOB	SAMPLE	DATAFILE	DF	ALS #	METHOD	UL INJ	CHEMIST	COMMENTS
	50ng DFTPP	X0600	1	1	DFTPP390	2.0	SLR	OK
	SSTD060X0316	X9437		2	X625A022	1.0		✓
	150	38		3				✓
	120	39		4				✓ curve OK
	30	40		5				✓
	05	41		6				✓
	3510 MIX 1	42		7				OK
	2	43		8				
	3	44		9				
	4	45		10				
	5	46		11				
	6	47		12				
	7	48		13				
	8	49		14				
	9	50		15				
	BLANK 1	51		16				
	2	52		17				

STANDARD	CODE
DFTPP	50856
CAL. STD.	5089-30 5089-49
IS MIX	AMP0934

REVIEWED AND APPROVED BY: _____
DATE: _____

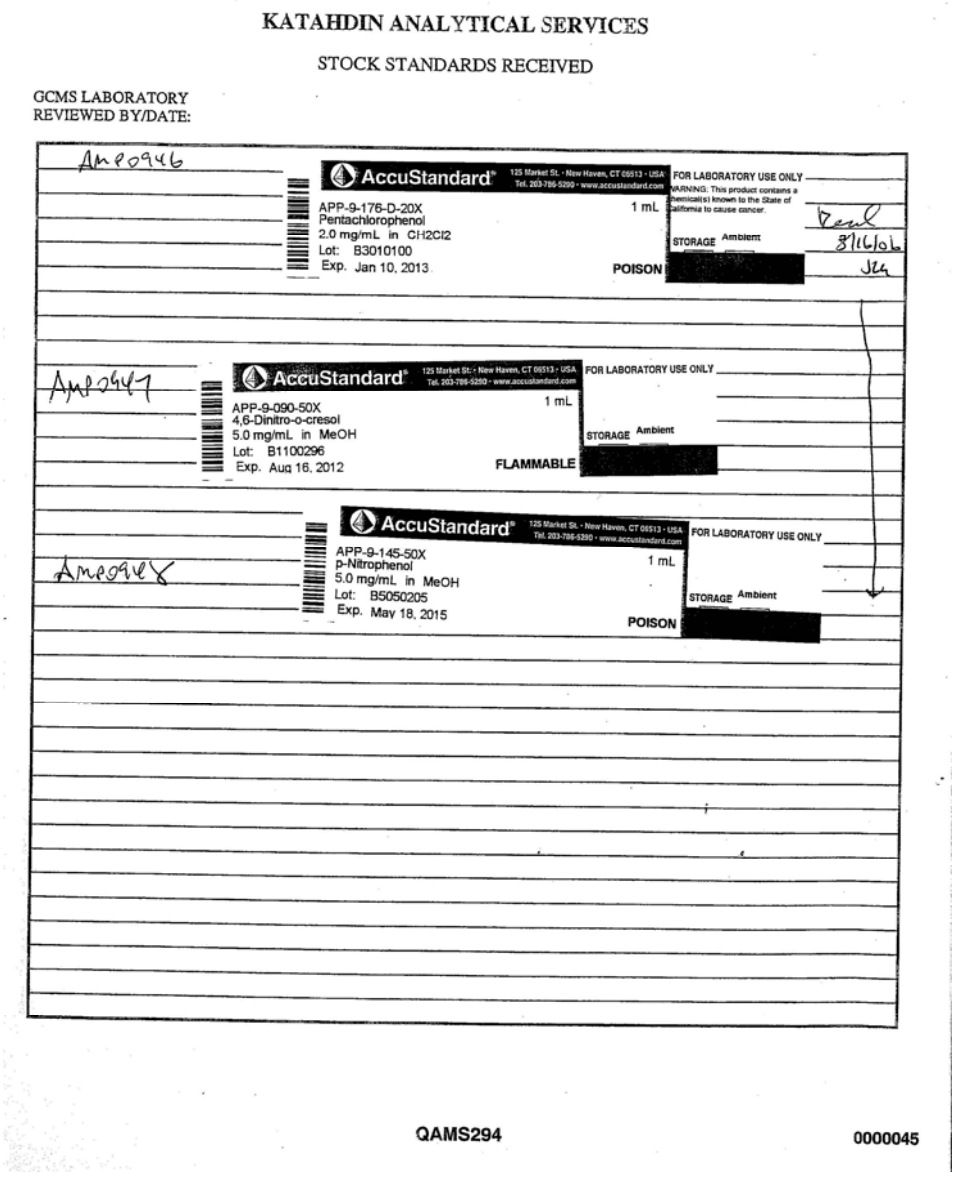
QAMS302

0000034

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY: SW 846 METHOD 8270 – Modified for Selected Ion Monitoring (SIM)

FIGURE 2

EXAMPLE OF GC/MS STANDARDS RECEIPT LOGBOOK ENTRY



KATAHDIN ANALYTICAL SERVICES

STOCK STANDARDS RECEIVED

GCMS LABORATORY
REVIEWED BY/DATE:

AM#0946

AccuStandard® 125 Market St. · New Haven, CT 06513 · USA Tel. 203-786-5290 · www.accustandard.com

APP-9-176-D-20X
Pentachlorophenol
2.0 mg/mL in CH2Cl2
Lot: B3010100
Exp. Jan 10, 2013

1 mL

FOR LABORATORY USE ONLY
WARNING: This product contains a chemical(s) known to the State of California to cause cancer.

STORAGE Ambient

POISON [redacted]

8/16/06

AM#0947

AccuStandard® 125 Market St. · New Haven, CT 06513 · USA Tel. 203-786-5290 · www.accustandard.com

APP-9-090-50X
4,6-Dinitro-o-cresol
5.0 mg/mL in MeOH
Lot: B1100296
Exp. Aug 16, 2012

1 mL

FOR LABORATORY USE ONLY

STORAGE Ambient

FLAMMABLE [redacted]

AM#0948

AccuStandard® 125 Market St. · New Haven, CT 06513 · USA Tel. 203-786-5290 · www.accustandard.com

APP-9-145-50X
p-Nitrophenol
5.0 mg/mL in MeOH
Lot: B5050205
Exp. May 18, 2015

1 mL

FOR LABORATORY USE ONLY

STORAGE Ambient

POISON [redacted]

QAMS294

0000045

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY: SW 846 METHOD 8270 – Modified for Selected Ion Monitoring (SIM)

FIGURE 3

EXAMPLE OF SVOA STANDARDS PREPARATION LOGBOOK ENTRY

GC/MS SVOA STANDARD PREP LOGBOOK

50863	8270 Stock (w/o MeOH)	3-15-06	7-7-06	JLh	AMP0844	8270 Megamix	300	2-22-07	4.2ml	150 ug/ml
					AMP0837	↓	300	3-15-07		
					AMP0911	APP IX #2	600	3-2-07		
					AMP0910	↓ 1	180	3-9-07		
					AMP0870	↓ 1	200	7-7-06		
					AMP0699	Organophosphorus	300	5-14-06		
					AMP0838	Benzoic Acid	↓	3-9-07		
					AMP0907	Hexachlorophene		2-22-07		
					AMP0906	Benzidine		3-9-07		
					AMP0836	3,3'-Dichlorobenzidine	↓	3-14-07		
					AMP0832	8270 Surr	150	3-9-07		
					50861	DEA	300	3-13-07		
					BY3E90	MeCl2	550	~		
50864	8270 level 1	3-15-06	7-7-06	JLh	50863	8270 Stock	70	7-7-06	1.05ml	10 ug/ml
					BY3E90	MeCl2	980			
50865	8270 level 2	3-15-06	7-7-06	JLh	50863	8270 Stock	150	7-7-06	0.90ml	25 ug/ml
					BY3E90	MeCl2	750			
50866	8270 level 3	3-15-06	7-7-06	JLh	50863	8270 Stock	600	7-7-06	1.8ml	50 ug/ml
					BY3E90	MeCl2	1200			
50867	8270 level 4	3-15-06	7-7-06	JLh	50864	8270 Stock	700	7-7-06	1.05ml	100 ug/ml
					BY3E90	MeCl2	350			

Reviewed by/Date:

CAMS284

0000057

TITLE: CLOSED-SYSTEM PURGE-AND-TRAP AND EXTRACTION FOR VOLATILE ORGANICS IN SOIL AND WASTE SAMPLES USING SW846 METHOD 5035

Prepared By: GC/MS Group Date: 7/98

Approved By:

Group Supervisor: [signature] Date: 011201

Operations Manager: [signature] Date: 1/15/01

QA Officer: [signature] Date: 1·23·01

General Manager: [signature] Date: 1/16/01

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
01 5035	Format changes, added pollution prevention, minor changes throughout	DM	1·23·01	12301
02 5035	Reorganized sections 4, 5, 6, 7 and 8.	MRC	07.02.04	07.02.04
03 5035	Editted section 6.4.3 to include the addition of 5mL of H2O to sample	LAD	020305	020305
04 5035	Balance weighs to 0.1g grammatical corrections formatting corrections	LAD	04/06	04/06
05	Added 3585 Reference. Sections 6.1.2.3, 6.4.3 and 7.2.2: changed 20mL to 5mL.	LAD	09/08	09/09

TITLE: **CLOSED-SYSTEM PURGE-AND-TRAP AND EXTRACTION FOR VOLATILE ORGANICS IN SOIL AND WASTE SAMPLES USING SW846 METHOD 5035**

Please acknowledge receipt of this standard operating procedure by signing and dating both of the spaces provided. Return the bottom half of this sheet to the QA Department.

I acknowledge receipt of copy ___ of document **SOP CA-214-05**, titled **CLOSED-SYSTEM PURGE-AND-TRAP AND EXTRACTION FOR VOLATILE ORGANICS IN SOIL AND WASTE SAMPLES USING SW846 METHOD 5035.**

Recipient: _____ Date: _____

KATAHDIN ANALYTICAL SERVICES, INC.
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy ___ of document **SOP CA-214-05**, titled **CLOSED-SYSTEM PURGE-AND-TRAP AND EXTRACTION FOR VOLATILE ORGANICS IN SOIL AND WASTE SAMPLES USING SW846 METHOD 5035.**

Recipient: _____ Date: _____

TITLE: **CLOSED-SYSTEM PURGE-AND-TRAP AND EXTRACTION FOR VOLATILE ORGANICS IN SOIL AND WASTE SAMPLES USING SW846 METHOD 5035**

1.0 SCOPE AND APPLICATION

This method describes a closed-system purge-and-trap process for the analysis of volatile organic compounds (VOCs) in solid materials (e.g., soils, sediments, and solid waste). While the method is designed for use on samples containing low levels of VOCs, procedures are also provided for collecting and preparing solid samples containing high concentrations of VOCs and for oily wastes. For these high concentration and oily materials, sample collection and preparation are performed using the procedures described here, and sample introduction is performed using the aqueous purge-and-trap procedure in Method 5030. These procedures may be used in conjunction with any appropriate determinative gas chromatographic procedure, including, but not limited to, Methods 8015, 8021, and 8260.

The low soil method utilizes a hermetically sealed sample vial, the seal of which is never broken from the time of sampling to the time of analysis. Since the sample is never exposed to the atmosphere after sampling, the losses of VOCs during sample transport, handling, and analysis are negligible. The applicable concentration range of the low soil method is dependent on the determinative method, matrix, and compound. However, it will generally fall in the 5.0 to 200 µg/kg range.

Procedures are included for preparing high concentration samples for purging by Method 5030. High concentration samples are those containing VOC levels of >200 µg/kg.

Procedures are also included for addressing oily wastes that are soluble in a water-miscible solvent by method 3585. These samples are also purged using Method 5030.

Method 5035 can be used for most volatile organic compounds that have boiling points below 200°C and that are insoluble or slightly soluble in water. Volatile, water-soluble compounds can be included in this analytical technique. However, quantitation limits (by GC or GC/MS) are approximately ten times higher because of poor purging efficiency.

The closed-system purge-and-trap equipment employed for low concentration samples is not appropriate for soil samples preserved in the field with methanol. Such samples should be analyzed using Method 5030 (see the note in Sec. 6.2.2).

1.1 Definitions

1.2 Responsibilities

This method is restricted to use by, or under the supervision of analysts experienced in the analyses using Methods 5030, 5035 and 3585. Each analyst must demonstrate and document their ability to generate acceptable results with this method. Refer to Katahdin SOP QA-805, current revision, "Personnel Training & Demonstration of Capability".

It is the responsibility of all Katahdin technical personnel involved in analysis of soils by method 5035 to read and understand this SOP, adhere to the procedures outlined, and to properly document their data in the appropriate lab notebook. Any deviations from the test or irregularities with the samples should also be recorded in the lab notebook and reported to the department manager or designated qualified data reviewer responsible for this data.

It is the responsibility of the Department Manager to oversee that members of their group follow this SOP, to ensure that their work is properly documented and to initiate periodic review of the associated logbooks.

1.3 Safety

Users of this procedure must be cognizant of inherent laboratory hazards, proper disposal procedures for contaminated materials and appropriate segregation of hazardous wastes. The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical should be treated as a potential health hazard. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Everyone involved with the procedure must be familiar with the MSDSs (material safety data sheets) for all the materials used in this procedure.

Each qualified analyst or technician must be familiar with Katahdin Analytical safety procedures and the Katahdin Hazardous Waste Management Plan and must follow appropriate procedures. These include the use of appropriate personal protective equipment (PPE) such as safety glasses, gloves and lab coats when working with chemicals or near an instrument and not taking food or drink into the laboratory. Each analyst should know the location of a respirator and all safety equipment. Each analyst shall receive a safety orientation from their department manager, or designee, appropriate for the job functions they will perform.

1.4 Pollution Prevention/Waste Disposal

Whenever possible, laboratory personnel should use pollution prevention techniques to address their waste generation. Refer to the current revision of the Katahdin Hazardous Waste Management Plan for further details on pollution prevention techniques.

After analysis, partially-filled VOA vials and sample jars are returned to the appropriate refrigerators to be disposed of in adherence with the Katahdin Hazardous Waste Management Plan and Safety Manual and SOP SD-903, Sample Disposal, current revision. Expired standards are lab packed, placed in the Katahdin hazardous waste storage area, and disposed of in accordance with this SOP SD-903.

Sample aliquots used for analysis are disposed of in accordance with SOP SD-903 and the Katahdin Hazardous Waste Management Plan and Safety Manual. The soil samples must be decanted and the soil fraction disposed of separately in compliance with Katahdin's disposal policies.

2.0 SUMMARY OF METHOD

2.1 Low concentration soil method - generally applicable to and soils and other solid samples with VOC concentrations in the range of 5.0 to 200 µg/kg. Volatile organic compounds (VOCs) are determined by collecting an approximately 5-g sample, weighed in the field at the time of collection, and placing it in a pre-weighed vial with a septum-sealed screw-cap (see Sec. 4) that already contains a stirring bar and a sodium bisulfate or organic-free laboratory reagent grade water preservative solution. If the samples are sent to the laboratory in an Encore sampling device, the laboratory extrudes the sample into this vial containing a stirring bar and a sodium bisulfate or organic-free laboratory reagent grade water preservative solution. The entire vial is then placed, unopened, into the instrument carousel. Immediately before analysis, organic-free laboratory reagent grade water, surrogates, and internal standards (if applicable) are automatically added without opening the sample vial. The vial containing the sample is heated to 40° and the volatiles purged into an appropriate trap using an inert gas combined with agitation of the sample. Purged components travel via a transfer line to a trap. When purging is complete, the trap is heated and backflushed with helium to desorb the trapped sample components into a gas chromatograph for analysis by an appropriate determinative method.

2.2 High concentration soil method - generally applicable to soils and other solid samples with VOC concentrations greater than 200 µg/kg. The sample introduction technique in Sec. 2.1 is not applicable to all samples, particularly those containing high concentrations (generally greater than 200 µg/kg) of VOCs which may overload either the volatile trapping material or exceed the working range of the determinative instrument system (e.g., GC/MS, GC/FID, GC/EC, etc.). In such instances, this method describes two sample collection options and the corresponding sample purging procedures.

2.2.1 The first option is to collect a bulk sample in a vial or other suitable container without the use of the preservative solution described in Sec. 2.1. A portion of that sample is removed from the container in the laboratory and is dispersed in a water-miscible solvent (e.g., methanol) to dissolve the volatile organic constituents. An aliquot of the solution is added to 20 mL of laboratory reagent grade water in a purge tube. Surrogates and internal standards (if applicable) are added to the solution, then purged using

Method 5030, and analyzed by an appropriate determinative method. Because the procedure involves opening the vial and removing a portion of the soil, some volatile constituents may be lost during handling.

2.2.2 The second option is to collect an approximately 5-g sample in a pre-weighed vial with a septum-sealed screw-cap (see Sec 4) that contains a known aliquot of a water-miscible organic solvent (e.g., methanol). An aliquot of the solution is added to 20 mL of laboratory reagent grade water in a purge tube. Surrogates and internal standards (if applicable) are added to the solution, then purged using Method 5030, and analyzed by an appropriate determinative method.

2.3 High concentration oily waste method - generally applicable to oily samples with VOC concentrations greater than 200 µg/kg that can be diluted in a water-miscible solvent. Samples that are comprised of oils or samples that contain significant amounts of oil present additional analytical challenges. This procedure is generally appropriate for such samples when they are soluble in a water-miscible solvent.

2.3.1 After demonstrating that a test aliquot of the sample is soluble in methanol, a separate aliquot of the sample is diluted in the appropriate solvent. An aliquot of the solution is added to 20 mL of laboratory reagent grade water in a purge tube, taking care to ensure that a floating layer of oil is not present in the purge tube. Internal standards (if applicable) and surrogates are added to the solution that is then purged using Method 5030 and analyzed by an appropriate determinative method.

2.3.2 Samples that contain oily materials that are not soluble in water-miscible solvents must be prepared in n-hexandecane according to Method 3585.

3.0 INTERFERENCES

3.1 Impurities in the purge gas and from organic compounds out-gassing from the plumbing ahead of the trap account for the majority of contamination problems. The analytical system must be demonstrated to be free from contamination under the conditions of the analysis by running method blanks. The use of non-polytetrafluoroethylene (non-PTFE) plastic coating, non-PTFE thread sealants, or flow controllers with rubber components in the purging device must be avoided, since such materials out-gas organic compounds which will be concentrated in the trap during the purge operation. These compounds will result in interferences or false positives in the determinative step.

3.2 Samples can be contaminated by diffusion of volatile organics (particularly methylene chloride and fluorocarbons) through the septum seal of the sample vial

during shipment and storage. A trip blank prepared from organic-free laboratory reagent grade water and carried through sampling and handling protocols serves as a check on such contamination.

3.3 Contamination by carryover can occur whenever high-concentration and low-concentration samples are analyzed in sequence. Where practical, samples with unusually high concentrations of analytes should be followed by an analysis of organic-free laboratory reagent grade water to check for cross-contamination. If the target compounds present in an unusually concentrated sample are also found to be present in the subsequent samples, the analyst must demonstrate that the compounds are not due to carryover. Conversely, if those target compounds are not present in the subsequent sample, then the analysis of organic-free laboratory reagent grade water is not necessary.

3.4 The laboratory where volatile analysis is performed should be free of solvents. The analytical and sample storage area should be isolated from all atmospheric sources of methylene chloride, otherwise random background levels will result. Since methylene chloride will permeate through PTFE tubing, all GC carrier gas lines and purge gas plumbing should be constructed of stainless steel or copper tubing. Laboratory workers' clothing previously exposed to methylene chloride fumes during common liquid/liquid extraction procedures can contribute to sample contamination. The presence of other organic solvents in the laboratory where volatile organics are analyzed will also lead to random background levels and the same precautions must be taken.

4.0 APPARATUS AND MATERIALS

4.1 Sample Containers

The specific sample containers required will depend on the purge-and-trap system to be employed (see Sec. 4.2). Our laboratory is equipped with Archon model 5100 purge and trap autosampler systems. A standard 40 ml VOA vial is used (e.g. ESS pre-cleaned certified 40 ml clear Type I borosilicate glass vials, open-top/polypropylene with 0.125 inch septa).

4.2 Purge-and-Trap System

The purge-and-trap system consists of a unit that automatically adds water, surrogates, and internal standards to a vial containing the sample, purges the VOCs using an inert gas stream while agitating the contents of the vial, and also traps the released VOCs for subsequent desorption into the gas chromatograph. The Archon systems at Katahdin meet the following criteria:

4.2.1 The purging device should be capable of accepting a vial sufficiently large to contain a 5-g soil sample plus a magnetic stirring bar and 10 mL of water. The device must be capable of heating a soil vial to 40°C and holding it at that temperature while the inert purge gas is allowed to pass through the sample. The device should also be capable of introducing at least 20 mL of organic-free laboratory reagent grade water into the sample vial while trapping the displaced headspace vapors. It must also be capable of agitating the sealed sample during purging, (e.g., using a magnetic stirring bar added to the vial prior to sample collection, sonication, or other means). The analytes being purged must be quantitatively transferred to an absorber trap. The trap must be capable of transferring the absorbed VOCs to the gas chromatograph (see 4.2.2).

4.2.2 A variety of traps and trapping materials may be employed with this method. The choice of trapping material may depend on the analytes of interest. Whichever trap is employed; it must demonstrate sufficient adsorption and desorption characteristics to meet the quantitation limits of all the target analytes for a given project and the QC requirements in Method 8000 and the determinative method. The most difficult analytes are generally the gases, especially dichlorodifluoromethane. The trap must be capable of desorbing the late eluting target analytes.

NOTE: Check the responses of the brominated compounds when using alternative charcoal traps (especially Vocarb 4000), as some degradation has been noted when higher desorption temperatures (especially above 240°C - 250°C) are employed. 2-Chloroethyl vinyl ether is degraded on Vocarb 4000 but performs adequately when Vocarb 3000 is used. The primary criterion, as stated above, is that all target analytes meet the sensitivity requirements for a given project.

4.2.2.1 The standard trap used in other EPA purge-and-trap methods is also acceptable. That trap is 25 cm long and has an inside diameter of at least 0.105 in. Starting from the inlet, the trap contains the equal amounts of the adsorbents listed below. It is recommended that 1.0 cm of methyl silicone-coated packing (35/60 mesh, Davison, grade 15 or equivalent) be inserted at the inlet to extend the life of the trap. If the analysis of dichlorodifluoromethane or other fluorocarbons of similar volatility is not required, then the charcoal can be eliminated and the polymer increased to fill 2/3 of the trap. If only compounds boiling above 35° are to be analyzed, both the silica gel and charcoal can be eliminated and the polymer increased to fill the entire trap.

4.2.2.1.1 2,6-Diphenylene oxide polymer - 60/80 mesh,

chromatographic grade (Tenax GC or equivalent).

4.2.2.1.2 Methyl silicone packing - OV-1 (3%) on Chromosorb-W, 60/80 mesh or equivalent.

4.2.2.1.3 Coconut charcoal - Prepare from Barnebey Cheney, CA-580-26, or equivalent, by crushing through 26 mesh screen.

4.2.2.2 Trapping materials other than those listed above also may be employed, provided that they meet the specifications in Sec. 4.2.3, below.

4.2.3 The desorber for the trap must be capable of rapidly heating the trap to the temperature recommended by the trap material manufacturer, prior to the beginning of the flow of desorption gas. Several commercial desorbers (purge-and-trap units) are available.

4.3 Syringe and Syringe Valves

4.3.1 25-mL glass hypodermic syringes with Luer-Lok (or equivalent) tip (other sizes are acceptable depending on sample volume used).

4.3.2 25-µL micro syringe with a 2 inch x 0.006 inch ID, 22° bevel needle (Hamilton #702N or equivalent).

4.3.3 Micro syringes - 10-, 100-µL.

4.3.4 Syringes - 0.5-, 1.0-, and 5-mL, gas-tight.

4.4 Miscellaneous

4.4.1 Glass vials

4.4.1.1 60-mL, septum-sealed, to collect samples for screening, dry weight determination.

4.4.1.2 40-mL, screw-cap, PTFE lined, septum-sealed. Examine each vial prior to use to ensure that the vial has a flat, uniform sealing surface.

4.4.2 Top-loading balance - Capable of accurately weighing to 0.1 g.

4.4.3 Glass scintillation vials - 20-mL, with screw-caps and PTFE liners, or glass culture tubes with screw-caps and PTFE liners, for dilution of oily waste samples.

4.4.4 Volumetric flasks - Class A, 10-mL and 100-mL, with ground glass stoppers.

4.4.5 2-mL glass vials, for GC autosampler - Used for oily waste samples extracted with methanol or PEG.

4.4.6 Spatula, stainless steel - narrow enough to fit into a sample vial.

4.4.7 Disposable Pasteur pipettes.

4.4.8 Magnetic stirring bars - PTFE- or glass-coated, of the appropriate size to fit the sample vials. Consult manufacturer's recommendation for specific stirring bars. Stirring bars may be reused, provided that they are thoroughly cleaned between uses. Consult the manufacturers of the purging device and the stirring bars for suggested cleaning procedures.

4.5 Field Sampling Equipment

4.5.1 EnCore™ sampler - (En Chem, Inc., 1795 Industrial Drive, Green Bay, WI 54302), or equivalent.

4.5.2 Alternatively, disposable plastic syringes with a barrel smaller than the neck of the soil vial may be used to collect the sample. The syringe end of the barrel is cut off prior to sampling. One syringe is needed for each sample aliquot to be collected.

4.5.3 Portable balance - For field use, capable of weighing to 0.01 g.

4.5.4 Balance weights - Balances employed in the field should be checked against an appropriate reference weight at least once daily, prior to weighing any samples, or as described in the sampling plan. The specific weights used will depend on the total weight of the sample container, sample, stirring bar, laboratory reagent grade water added, cap, and septum.

5.0 REAGENTS

5.1 Organic-free laboratory reagent grade water - All references to water in this method refer to organic-free laboratory reagent grade water.

5.2 Methanol, CH_3OH - purge-and-trap quality or equivalent. Store away from other solvents.

5.3 Sodium bisulfate, $NaHSO_4$ - ACS reagent grade or equivalent.

5.4 Polyethylene glycol (PEG), $H(OCH_2CH_2)_nOH$ – free of interferences at the detection limit of the target analytes.

5.5 See the determinative method for guidance on internal standards and surrogates to be employed in this procedure.

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

The low concentration portion of this method employs sample vials that are filled and weighed in the field and never opened during the analytical process. As a result, sampling personnel should be equipped with a portable balance capable of weighing to 0.01 g.

6.1 Preparation of sample vials

The specific preparation procedures for sample vials depend on the expected concentration range of the sample, with separate preparation procedures for low concentration soil samples and high concentration soil and solid waste samples. Sample vials should be prepared in a fixed laboratory or other controlled environment, sealed, and shipped to the field location. Gloves should be worn during the preparation steps.

Because volatile organics will partition into the headspace of the vial from the aqueous solution and will be lost when the vial is opened, surrogates, matrix spikes, and internal standards should only be added to the vials back in the laboratory, either manually by puncturing the septum with a small-gauge needle or automatically by the sample introduction system, just prior to analysis.

6.1.1 Low concentration soil samples

Sodium bisulfate preservation is used in the preparation of vials used in the collection of low concentration soil samples to be analyzed by the closed-system purge-and-trap equipment described in Method 5035.
Water and subsequent freezing preparation of vials is used in the collection of low concentration soil samples known to contain carbonate minerals which may effervesce upon contact with an acidic preservation solution and which are to be analyzed by the closed-system purge-and-trap equipment

described in Method 5035. This type of preservation is typically done in the lab after Encore samplers are received from the field

6.1.1.1 Add a clean magnetic stirring bar to each clean vial.

6.1.1.2 The preservative is added to each vial prior to shipping the vial to the field. Add 20 mL of 20% sodium bisulfate solution or 20 mL of water to the vial and seal the vial with the screw-cap and septum seal.

6.1.1.3 Affix a label to each vial. This eliminates the need to label the vials in the field and assures that the tare weight of the vial includes the label. (The weight of any markings added to the label in the field is negligible). It is important that labels and tape not cover the junction of the screw top and vial. Labels and tape must also be applied smoothly (i.e. no wrinkles) to prevent autosampler failures.

6.1.1.4 Weigh the prepared vial to the nearest 0.1 g and record it on the label.

6.1.2 High concentration soil samples in methanol:

6.1.2.1 When high concentration samples are collected without a preservative, a variety of sample containers may be employed, including 40-mL glass vials with septum seals (see Sec. 4.4).

6.1.2.2 The following steps apply to the preparation of vials used in the collection of high concentration soil samples to be preserved in the field with methanol and analyzed by the aqueous purge-and-trap equipment described in Method 5030.

6.1.2.3 Add 5 mL of methanol to each vial.

6.1.2.4 Seal the vial with the screw-cap and septum seal.

6.1.2.5 Affix a label to each vial. This eliminates the need to label the vials in the field and assures that the tare weight of the vial includes the label. (The weight of any markings added to the label in the field is negligible).

6.1.2.6 Weigh the prepared vial to the nearest 0.01 g and record it on the label.

NOTE: Vials containing methanol should be weighed a second time on the day that they are to be used. Vials found to have lost methanol (reduction in weight of >0.01 g) should not be used for sample collection.

6.1.3 Oily waste samples

When oily waste samples are known to be soluble in methanol, sample vials may be prepared as described in Sec. 6.1.2.2, using the appropriate solvent. However, when the solubility of the waste is unknown, the sample should be collected without the use of a preservative, in a vial such as that described in Sec. 6.1.2.1.

6.2 Sample collection

Collect the sample according to the procedures outlined in the sampling plan. As with any sampling procedure for volatiles, care must be taken to minimize the disturbance of the sample in order to minimize the loss of the volatile components. Several techniques may be used to transfer a sample to the relatively narrow opening of the low concentration soil vial. These include devices such as the EnCore™ sampler, the Purge-and-Trap Soil Sampler™ , and a cut plastic syringe. Always wear gloves whenever handling the tared sample vials.

6.3 Sample handling and shipment

All samples for volatiles analysis should be cooled to approximately 4°C, packed in appropriate containers, and shipped to the laboratory on ice, as described in the sampling plan. Samples should be shipped on the day of sampling if at all possible.

6.4 Sample storage

6.4.1 Once in the laboratory, store samples at 4°C until analysis. The sample storage area should be free of organic solvent vapors.

6.4.2 All samples should be analyzed as soon as practical, and within the designated holding time from collection. Samples not analyzed within the designated holding time must be noted and the data are considered minimum values.

6.4.3 When the low concentration samples are strongly alkaline or highly calcareous in nature, the sodium bisulfate preservative solution may not be strong enough to reduce the pH of the soil/water solution to below 2. Therefore, when low concentration soils to be sampled are known or suspected to be strongly alkaline or highly calcareous, additional steps may

be required to preserve the samples. Such steps include: addition of larger amounts of the sodium bisulfate preservative to non-calcareous samples, or the addition of 5 mL of water and storage at -10° (taking care not to fill the vials so full that the expansion of the water in the vial breaks the vial), or significantly reducing the maximum holding time for low concentration soil samples.

7.0 PROCEDURES

This section describes procedures for the low concentration soil method, the high concentration soil method, and the procedure for oily waste samples. High concentration samples are to be introduced into the GC system using Method 5030. Oily waste samples are to be introduced into the GC system using Method 5030 if they are soluble in a water-miscible solvent, or using Method 3585 if they are not.

For the high concentration soil and oily waste samples, the surrogate compounds may either be spiked into the solvent at the time of extraction or the laboratory reagent grade water containing an aliquot of the extract prior to analysis.

7.1 Low concentration soil method (Approximate concentration range of 5 to 200 µg/kg -the concentration range is dependent upon the determinative method and the sensitivity of each analyte.)

7.1.1 Archon Operation

Prior to using this introduction technique for any GC or GC/MS method, the system must be calibrated by the analytical method to be used. When a GC/MS method is used, internal standard calibration is employed.

7.1.1.1 Establish the purge-and-trap instrument operating conditions. Adjust the instrument to inject 10 mL of water, to heat the sample to 40°C, and to hold the sample at 40°C for 1.5 minutes before commencing the purge process, or as recommended by the instrument manufacturer.

7.1.1.2 Carry out the purge-and-trap procedure as outlined in Secs. 7.1.2. to 7.1.4.

7.1.2 Sample purge-and-trap

This method is designed for a 5-g sample size, but smaller sample sizes may be used. The soil vial is hermetically sealed at the sampling site, and MUST remain sealed in order to guarantee the integrity of the sample.

Gloves must be worn when handling the sample vial since the vial has been tared. If any soil is noted on the vial or cap, it must be carefully removed prior to weighing. Weigh the vial and contents to the nearest 0.01 g, even if the sample weight was determined in the field, and record this weight. This second weighing provides a check on the field sampling procedures and provides additional assurance that the reported sample weight is accurate. Data users should be advised on significant discrepancies between the field and laboratory weights.

7.1.2.1 Remove the sample vial from storage and allow it to warm to room temperature. Shake the vial gently, to ensure that the contents move freely and that stirring will be effective. Place the sample vial in the instrument carousel according to the manufacturer's instructions.

7.1.2.2 Without disturbing the hermetic seal on the sample vial, add 10 mL of organic-free laboratory reagent grade water, the internal standards, and the surrogate compounds. This is carried out either manually or using the automated sampler. Other volumes of organic-free laboratory reagent grade water may be used. However, it is imperative that all samples, blanks, and calibration standards have exactly the same final volume of organic-free laboratory reagent grade water. Prior to purging, heat the sample vial to 40°C for 1.5 minutes, or as described by the manufacturer.

7.1.2.3 For the sample selected for matrix spiking, add the matrix spiking solution described in Sec. 5.0 of Method 5000, either manually, or automatically, following the manufacturer's instructions.

7.1.2.4 Purge the sample with helium or another inert gas at a flow rate of up to 40 mL/minute (the flow rate may vary from 20 to 40 mL/min, depending on the target analyte group) for 11 minutes while the sample is being agitated with the magnetic stirring bar or other mechanical means. The purged analytes are allowed to flow out of the vial through a transfer line to a trap packed with suitable sorbent materials.

7.1.3 Sample Desorption

7.1.3.1 Non-cryogenic interface - After the 11 minute purge, place the purge-and-trap system in the desorb mode and preheat the trap to 245°C without a flow of desorption gas. Start the flow of desorption gas at 10 mL/minute for about four minutes. Begin the temperature program of the gas chromatograph and start data acquisition.

7.1.4 Trap Reconditioning

After desorbing the sample for 4 minutes, recondition the trap by returning the purge-and-trap system to the purge mode. Maintain the trap temperature at 245°C (or other temperature recommended by the manufacturer of the trap packing materials). After approximately 10 minutes, turn off the trap heater and halt the purge flow through the trap. When the trap is cool, the next sample can be analyzed.

7.2 High concentration method for soil samples with concentrations generally greater than 200 µg/kg.

The high concentration method for soil is based on a solvent extraction. A solid sample is either extracted or diluted, depending on sample solubility in a water-miscible solvent. An aliquot of the extract is added to organic-free laboratory reagent grade water containing surrogates, internal and matrix spiking standards (added manually or by the autosampler), purged according to Method 5030, and analyzed by an appropriate determinative method. The specific sample preparation steps depend on whether or not the sample was preserved in the field. Samples that were not preserved in the field are prepared using the steps below, beginning at Sec. 7.2.1. If solvent preservation was employed in the field, then the preparation begins with Sec. 7.2.4.

7.2.1 When the high concentration sample is not preserved in the field, the sample consists of the entire contents of the sample container. Do not discard any supernatant liquids. Remove a representative aliquot with a spatula.

7.2.2 For soil and solid waste samples that are soluble in methanol, add 5.0 g (wet weight) of sample to a tared 40-mL VOA vial using a calibrated (refer to Katahdin SOP, CA-102, Balance Calibration) top loading balance. Record the weight to 0.1 g. Add 5 mL of methanol to the vial containing the sample and shake for two minutes.

NOTE: The steps in Secs. 7.2.1, 7.2.2, and 7.2.3 must be performed rapidly and without interruption to avoid loss of volatile organics. These steps must be performed in a laboratory free from solvent fumes.

7.2.4 For soil and solid waste samples that were collected in methanol or PEG, weigh the vial to 0.01 g as a check on the weight recorded in the field.

7.2.5 For each new lot of methanol, add an appropriate aliquot of the methanol to 20 mL of organic-free laboratory reagent grade water and analyze by Method 5030 in conjunction with the appropriate determinative method.

Proceed to Sec. 7.0 in Method 5030 and follow the procedure for purging high concentration samples.

7.3 High concentration method for oily waste samples

This procedure for the analysis of oily waste samples involves the dilution of the sample in methanol or PEG. However, care must be taken to avoid introducing any of the floating oil layer into the instrument. A portion of the diluted sample is then added to 5.0 mL of organic-free laboratory reagent grade water, purged according to Method 5030, and analyzed using an appropriate determinative method.
The specific sample preparation steps depend on whether or not the sample was preserved in the field. Samples that were not preserved in the field are prepared using the steps below, beginning at Sec. 7.3.2. If methanol preservation was employed in the field, then the preparation begins with Sec. 7.3.4.

7.3.1 For oily samples that are not soluble in methanol or PEG (including those samples consisting primarily of petroleum or coking waste), dilute or extract with hexadecane and shake for two minutes.

7.3.2 For oily samples that are soluble in methanol if the waste was not preserved in the field, tare a 10-mL volumetric flask, or a VOA vial, weigh 1 g (wet weight) of the sample into the tared vessel and add 10.0 mL methanol or PEG with a calibrated syringe. If a vial is used instead of a volumetric flask, it must be calibrated prior to use. This operation must be performed prior to opening the sample vial and weighing out the aliquot for analysis. Invert the vial a minimum of three times to mix the contents.

7.3.4 If the sample was collected in the field in a vial containing methanol or PEG, weigh the vial to 0.1 g as a check on the weight recorded in the field, and proceed with Sec. 7.3.5.

7.3.5 Regardless of how the sample was collected, the target analytes are extracted into the solvent along with the majority of the oily waste (i.e., some of the oil may still be floating on the surface). If oil is floating on the surface, transfer 1 to 2 mL of the extract to a clean GC vial using a Pasteur pipet. Ensure that no oil is transferred to the vial.

7.3.6 Add an appropriate aliquot of the methanol or PEG to 5.0 mL of organic-free laboratory reagent grade water and analyze by Method 5030 in conjunction with the appropriate determinative method. Proceed to Sec. 7.0 in Method 5030 and follow the procedure for purging oily waste samples.

7.4 Determination of % Dry Weight

If results are to be reported on a dry weight basis, it is necessary to determine the dry weight of the sample. Refer to Katahdin SOP, CA-717, for determination of % dry weight.

NOTE: It is highly recommended that the dry weight determination only be made after the analyst has determined that no sample aliquots will be taken from the 60-mL vial for high concentration analysis. This is to minimize loss of volatiles and to avoid sample contamination from the laboratory atmosphere. There is no holding time associated with the dry weight determination. Thus, this determination can be made any time prior to reporting the sample results, as long as the vial containing the additional sample has remained sealed and properly stored.

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

8.1 Before processing any samples, the analyst should demonstrate through the analysis of an organic-free laboratory reagent grade water method blank that all glassware and reagents are interference free. Each time a set of samples is extracted, or there is a change in reagents, a method blank should be processed as a safeguard against chronic laboratory contamination. The blank samples should be carried through all stages of the sample preparation and measurement.

8.2 Initial Demonstration of Proficiency - Each laboratory must demonstrate initial proficiency with each sample preparation and determinative method combination it utilizes, by generating data of acceptable accuracy and precision for target analytes in a clean matrix. The laboratory must also repeat this demonstration whenever new staff are trained or significant changes in instrumentation are made.

8.3 Sample Quality Control for Preparation and Analysis - See the appropriate analytical method to follow to demonstrate acceptable continuing performance on each set of samples to be analyzed. These include the method blank, either a matrix spike/matrix spike duplicate or a matrix spike and duplicate sample analysis, a laboratory control sample (LCS), and the addition of surrogates to each sample and QC sample.

9.0 METHOD PERFORMANCE

Refer to appropriate analytical method.

TITLE: **CLOSED-SYSTEM PURGE-AND-TRAP AND EXTRACTION FOR VOLATILE ORGANICS IN SOIL AND WASTE SAMPLES USING SW846 METHOD 5035**

10.0 APPLICABLE DOCUMENTS/REFERENCES

"Test Methods for the Evaluation of Solid Waste: Physical/Chemical Methods," Method 5035, SW-846, USEPA, Revision III, June, 1997.

"Test Methods for the Evaluation of Solid Waste: Physical/Chemical Methods," Method 5035A, SW-846, USEPA, Revision III, June, 1997.

"Test Methods for the Evaluation of Solid Waste: Physical/Chemical Methods," Method 3585, SW-846, USEPA, Revision IIIB, Nov., 2004.

Archon Operator's Manual

LIST OF TABLES AND FIGURES

TITLE: **CLOSED-SYSTEM PURGE-AND-TRAP AND EXTRACTION FOR VOLATILE ORGANICS IN SOIL AND WASTE SAMPLES USING SW846 METHOD 5035**

TABLE 1

SUMMARY OF METHOD MODIFICATIONS

TOPIC	KATAHDIN SOP CA-214-05	METHOD 5035, current revision
Apparatus/Materials		
Reagents		
Sample preservation/ handling		
Procedures	(1) Use methanol prep for all high concentration soils. (2) For high concentration soils, leave all extract in the vial with the soil for storage.	(1) For high concentration soils from an unknown source, perform a solubility test. (2) For high concentration soils, pipet approximately 1 mL of extract into a GC vial for storage.

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY CAPILLARY COLUMN GC/MS: SW 846 METHOD 8270D

Prepared By: Semivolatile Group Date: 02.11.09

Approved By:

Department Manager: [signature] Date: 2-11-09

Operations Manager: Deborah J. Kadeau Date: 2.11.09

QA Officer: Leslie Dimond Date: 02.10.09

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
01	Updated to reflect DoD QSM version 4.1 compliance and new standard preparation procedures.	EH	08/09	08/09

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY CAPILLARY COLUMN GC/MS: SW 846 METHOD 8270

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY CAPILLARY COLUMN GC/MS: SW 846 METHOD 8270D.

Please acknowledge receipt of this standard operating procedure by signing and dating both of the spaces provided. Return the bottom half of this sheet to the QA Department.

I acknowledge receipt of copy ___ of document SOP CA-226-01, titled "Analysis of Semivolatile Organic Compounds by Capillary Column GC/MS: SW 846 Method 8270D".

Recipient: _____ Date: _____

KATAHDIN ANALYTICAL SERVICES, INC.
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy ___ of document SOP CA-226-01, titled "Analysis of Semivolatile Organic Compounds by Capillary Column GC/MS: SW 846 Method 8270D".

Recipient: _____ Date: _____

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY CAPILLARY COLUMN GC/MS: SW 846 METHOD 8270D.

1.0 SCOPE AND APPLICATION

The purpose of this SOP is to describe the procedures utilized by Katahdin Analytical Services, Inc. laboratory personnel to prepare and analyze water and soil sample extracts for semivolatile organics by EPA SW-846 Method 8270D.

In order to maintain consistency in data quality, this SOP consolidates all aspects of the analyses in one working document, to be revised as necessary.

1.1 Definitions:

ANALYTICAL BATCH: 20 or fewer samples which are analyzed together with the same method sequence and the same lots of reagents and with the manipulations common to each sample within the same time period or in continuous sequential time periods.

METHOD BLANK (laboratory reagent blank): An artificial sample designed to determine if method analytes or other interferences are present in the laboratory environment, the reagents, or the apparatus. For aqueous samples, laboratory reagent grade water is used as a blank matrix; for soil samples, baked organic-free sand is used as a blank matrix. The blank is taken through the appropriate steps of the process.

CALIBRATION CHECK: Verification of the ratio of instrument response to analyte amount; a calibration check is done by analyzing for analyte standards in an appropriate solvent. Calibration check solutions are made from a stock solution, which is different from the stock used to prepare standards.

INDEPENDANT CALIBRATION STANDARD: A solution prepared from a stock standard solution independent of the standard that is used to calibrate the instrument. Analyzed immediately after calibration,

CALIBRATION STANDARD (WORKING STANDARD): A solution prepared from the stock standard solution, which is used to calibrate the instrument response with respect to analyte concentration.

LABORATORY CONTROL SAMPLE (LCS): A blank that has been spiked with the analyte(s) from an independent source, and is analyzed exactly like a sample. Its purpose is to determine whether the methodology is in control, and whether the laboratory is capable of making accurate and precise measurements. The matrix used should be phase matched with the samples and well characterized.

MATRIX SPIKE/MATRIX SPIKE DUPLICATE (MS/MSD): Predetermined quantities of stock solutions of certain analytes are added to a sample matrix prior to sample extraction and analysis. Samples are split into duplicates, spiked and analyzed.

Percent recoveries are calculated for each of the analytes detected. The relative percent difference between the samples is calculated and used to assess analytical precision.

STANDARD CURVE (CALIBRATION CURVE): A curve that plots concentration of known analyte standard versus the instrument response to the analyte.

STOCK STANDARD SOLUTION: A concentrated solution containing a single certified standard that is a method analyte, or a concentrated solution of a single analyte prepared in the laboratory with an assay reference compound. Stock standard solutions are used to prepare calibration standards.

SURROGATES: Organic compounds which are similar to analytes of interest in chemical composition, extraction and chromatography, but which are not normally found in environmental samples. These compounds are spiked into all blanks, standards, samples and spiked samples prior to analysis. Percent recoveries are calculated for each surrogate.

TARGET: A software system that combines full processing, reporting and comprehensive review capabilities, regardless of chromatographic vendor and data type.

TARGET DB: An oracle database used to store and organize all Target data files.

QUICKFORMS: A laboratory reporting software for Target and Target DB. The QuickForms report module for Target is preconfigured with generalized forms and US EPA CLP report forms and disk deliverables, which can be customized.

1.2 Responsibilities

This method is restricted to use by, or under the supervision of analysts experienced in the analysis of semivolatile organic compounds by EPA Method 8270D. Each analyst must demonstrate and document their ability to generate acceptable results with this method. Refer to Katahdin SOP QA-805, “Personnel Training & Documentation of Capability,” current revision.

It is the responsibility of all Katahdin technical personnel involved in analysis of semivolatiles by Method 8270D to read and understand this SOP, to adhere to the procedures outlined, and to properly document their data in the appropriate lab notebook. Any deviations from the test or irregularities with the samples should also be recorded in the lab notebook and reported to the Department Manager or designated qualified data reviewer responsible for this data.

It is the responsibility of the Department Manager to oversee that members of their group follow this SOP, to ensure that their work is properly documented and to initiate periodic review of the associated logbooks.

1.3 Safety

Users of this procedure must be cognizant of inherent laboratory hazards, proper disposal procedures for contaminated materials and appropriate segregation of hazardous wastes. The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical should be treated as a potential health hazard. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Everyone involved with the procedure must be familiar with the MSDSs (material safety data sheets) for all the materials used in this procedure.

Each qualified analyst or technician must be familiar with Katahdin Analytical Environmental Health and Safety Manual including the Katahdin Hazardous Waste Plan and must follow appropriate procedures. These include the use of appropriate personal protective equipment (PPE) such as safety glasses, gloves and lab coats when working with chemicals or near an instrument and not taking food or drink into the laboratory. Each analyst should know the location of all safety equipment. Each analyst shall receive a safety orientation from their Department Manager, or designee, appropriate for the job functions they will perform.

1.4 Pollution Prevention/Waste Disposal

Whenever possible, laboratory personnel should use pollution prevention techniques to address their waste generation. Refer to the current revision of the Katahdin Hazardous Waste Management Program for further details on pollution prevention techniques.

After analysis, autosampler vials containing sample extracts in methylene chloride are returned to the SVOA hood, and the contents transferred to a labeled waste container. The contents of this container are disposed of in accordance with the Katahdin Hazardous Waste Management Plan and Safety Manual and SOP SD-903, “Sample Disposal,” current revision. Expired standards are lab packed, placed in the Katahdin hazardous waste storage area, and disposed of in accordance with this SOP.

2.0 SUMMARY OF METHOD

The process involves the extraction of semivolatiles from a sample using an appropriate solvent followed by clean up steps (where applicable) and concentration of the extract (refer to Katahdin SOP CA-502, “Preparation Of Aqueous Samples For Extractable Semivolatile Analysis”, SOP CA-512, “Preparation Of Sediment/Soil Samples By Sonication Using Method 3550 For Subsequent Extractable Semi-Volatiles Analysis” and SOP CA-526, “Preparation Of Sediment/Soil Samples By Soxhlet Extraction Using Method 3540 For Subsequent Extractable Semivolatile Analysis”). An aliquot of the final extract is injected into the gas chromatograph for compound separation by capillary column, followed by the

electron impact mass spectrometer for identification and quantitation.

Target and surrogate compounds are identified and compared to the mass spectra obtained from the analysis of standard solutions containing the same compounds. A relative response factor is established for each target compound and surrogate against an internal standard during the most recent initial or continuing calibrations. The identified compound is then quantitated using the relative response factor, the amount of internal standard in the sample, the initial volume of sample, and any other factors, such as dilutions.

3.0 INTERFERENCES

Interfering contamination may occur when a sample containing low concentrations of SVOCs is analyzed immediately after a sample containing high concentrations of SVOCs. Any samples that have suspected carryover must be reanalyzed.

4.0 APPARATUS AND MATERIALS

4.1 GC: Hewlett Packard 5890 and/or 6890

4.2 Mass Spectrometers (MS): HP5973, HP5972 and/or HP5970

4.3 Helium: Carrier gas for routine applications. All carrier gas lines must be constructed from stainless steel or copper tubing; non-polytetrafluoroethylene (non-PTFE) thread sealant or flow controllers with rubber component are not to be used.

4.4 Autosamplers: HP 7673As and HP 7683s

4.5 Hamilton syringes: 2.00 uL to 10 mL

4.6 Volumetric glassware: Grade A or equivalent

4.7 Columns: DB-5MS 30m, 0.25mm I.D., 25um film thickness, columns (J&W Scientific) or equivalent.

4.8 Acquisition System: The acquisition system must be interfaced to the MS and allow continuous acquisition of data throughout the duration of the chromatographic program. It must permit, at a minimum, the output of time vs. intensity (peak height or peak area). Hewlett Packard Chemstation or equivalent.

4.9 Data System: The Target software is used for processing data and generating forms.

4.10 1.8 mL vials with 350uL inserts

4.11 Crimp tops with Teflon lined septa

5.0 REAGENTS

5.1 J.T. Baker Ultra Resi-Analyzed methylene chloride (or equivalent)

5.2 Purge and trap grade methanol

5.3 Standards: Stock standards and working standards are received and recorded in accordance with SOP CA-106 “Standard Preparation and Documentation”.

5.3.1 The expiration date for all standards is one year from date of opening the ampule. If the manufacturer’s expiration date is before this one year date, the manufacturer’s expiration must be followed. New standards must be opened if degradation is observed.

5.3.2 Secondary dilution standards

The standards are prepared on an as needed basis (but not less than every 6 months) and stored in screw cap amber bottles with Teflon liners in the BNA standards freezer between uses. Standards prepared from various stock solutions must always use the first expiration date of any of the solutions used for preparation.

5.3.2.1 Calibration Mix – Prepare a standard stock mix that contains those compounds commonly considered 8270 and those compounds commonly considered Appendix IX compounds. The compound dinoseb should <u>not</u> be added to this stock as it is only available in methanol. This will be added separately to each calibration level. Use Table 3 as a guide. The stock should be prepared at 125 ug/mL.

5.3.2.2 Independent Calibration Verification (ICV) Standard – From a source other than that used to make the calibration standards, prepare separate standards mixes (A and B) such that Standard Mix A contains those compounds commonly considered 8270 and Standard B Mix contains those compounds commonly considered Appendix IX compounds. Use Table 3 as a guide. Each stock should be prepared at 100 ug/mL.

5.3.2.3 DFTPP Solution – Prepare standard in methylene chloride containing DFTPP, Pentachlorophenol, Benzidine and DDT at a final concentration of 25 ug/mL.

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY CAPILLARY COLUMN GC/MS: SW 846 METHOD 8270D.

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

All semivolatile sample extracts should be refrigerated until analysis. Extracts must be analyzed within forty days following the date of extraction.

7.0 PROCEDURES

7.1 NAMING AND CODING CONVENTIONS FOR ANALYTICAL STANDARDS – Used in accordance with SOP CA-106 “Standard Preparation and Documentation”.

7.2 COMPUTER (DATA SYSTEM) CONVENTIONS -

Conventions for all instruments are as follows:

Sub-Directory for data acquisition and storage: C:\HPCHEM\1\DATA

Tune file: DFTPP.U

Method files: L8270CXX.M (all samples and standards)

Where:
XX = the calibration number in chronological order
L = instrument ID (R, U, or G)

DFTPP tuning acquisition: DFTPP390.M

NOTE: All acquisition parameters must be identical for L8270CXX.M and DFTPP390. M.

Data Files: L_ _ _ _.D, where _ _ _ _ is a number in chronological order from 0001 to 9999 and L is the instrument ID (R, U, or G). This file also contains the Quantitation output file.

Data Files for DFTPP: LD_ _ _.D, where _ _ _ is a number in chronological order from 001 to 999 and L is the instrument ID (R, U, or G).

7.3 INSTRUMENT SPECIFIC PROCEDURES

It is the policy of the GC/MS group that all data be acquired in the batch mode. The following items must be checked prior to data acquisition in the batch mode:

- Ensure that the proper sequence and tune files are being used.
- Check the autosampler syringe (Is it clean? Does the plunger move freely? etc.), its alignment and make sure the solvent rinse vial is full. Ensure that the knurled nut holding the top of the syringe plunger is tight.

- Look at the batch to be analyzed and check the following:
  - Make sure that the data files are in numerical order with no duplication and that the method file is the same as that used for ICAL or Continuing Calibration analysis.
  - Bottle numbers match with the numbers on the autosampler tray.

After the batch has been deemed free of errors, start the batch by using the "Position and run" command under the SEQUENCE menu in MSTop.

7.4 INSTRUMENT TUNING - Prior to the analysis of any calibration standards, blanks or samples, the GC/MS system must be shown to meet the mass spectral key ion and ion abundance criteria for decafluorotriphenylphosphine (DFTPP) tabulated below. Pentachlorophenol, benzidine and DDT are also present in this standard.

Mass	Criteria
51	30 to 60% of mass 198
68	< 2% of mass 69
70	< 2% of mass 69
127	40-60% of mass 198
197	< 1% of mass 198
198	base peak, 100 % relative abundance
199	5-9% of mass 198
275	10-30% of mass 198
365	> 1% of mass 198
441	present but less than mass 443
442	> 40% of mass 198
443	17-23% of mass 442

All ion abundances must be normalized to m/z 198, the nominal base peak.

The following are the GC/MS operating conditions for injection of DFTPP.

Initial column temperature hold	140°C for 3 minutes
Column temperature program	140-275°C at 15 degrees/minute
Final column temperature hold	275°C
Injection port temperature	280°C
Transfer line/source temperature	285°C
Injector - splitless, valve time	0.18 minutes
EPC	inlet B
Constant flow	ON
Constant flow pressure	10psi
Constant flow temperature	30°C
Vacuum comp.	ON

Run time	10-12 minutes
Scan start time	5.0-6.0 minutes
Sample volume	2.0 uL of 25 ng/uL DFTPP solution
Carrier gas	helium at approximately 60 mL/minute
Mass range	35 to 500 amu
Number of A/D samples	4
GC Peak threshold	500 counts
Threshold	10 counts

Set up the run on the Enviroquant system using "Edit Sample Log Table". For a more detailed explanation of the Enviroquant software, consult the appropriate manual, Department Manager, or senior chemist within the GC/MS group.

When the DFTPP has concluded, the run must be evaluated to determine if sample analysis can proceed. The chromatography and the ion ratios must be examined. The DFTPP run is processed using the current algorithms in the Target software.

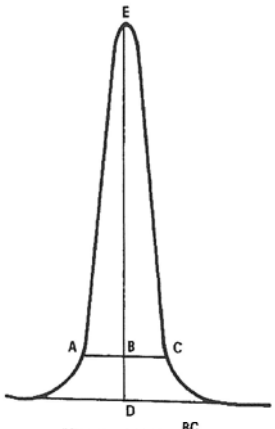
If the results indicate the system does not meet acceptance criteria, the GC/MS must be manually tuned. Once the manual tune procedure is completed, DFTPP must be re-injected and reevaluated. If the instrument still does not meet criteria, notify your Department Manager. Under no circumstances should calibration proceed if the instrument DFTPP is not in criteria.

The DFTPP tuning standard should also be used to assess the column performance and injection port inertness. Calculate the degradation of DDT to DDE and DDD; it should not exceed 20%. Benzidine and pentachlorophenol should be present at their normal responses, and should not exceed a tailing factor of 2 given by the followint equation:

$$\text{Tailing Factor} = \frac{BC}{AC}$$

.Where: AC = the width at 10% height
DE = height of the peak
B = the height at 10% of DE

Example:



In order to document the performance of benzidine, pentachlorophenol and DDT, the following procedure must be followed. At the PC, which operates the instrument, load the method TUNETAIL.M into the ENVDA screen. Go into the quant drop down menu and select *calculate/generate report*. When that finishes, select *Qedit quant result.* Each compound can now be evaluated. Double click on benzidine and select *ChromEval* and then *Evaluate tailing*. Follow the instructions given on the screen to evaluate tailing. Send the report to the printer. Repeat the procedure for pentachlorophenol. Repeat the procedure for DDT, selecting *Evaluate degradation*. Follow the instructions given on the screen and then send the report to the printer. The report should be filed with the tune raw data.

The DFTPP solution must be analyzed once at the beginning of each twelve hour period during which standards and/or samples are analyzed. The 12 hour time period for GC/MS system begins at the moment of injection of the DFTPP analysis. The time period ends after twelve hours has elapsed according to the system clock. The last injection must be accomplished prior to the expiration of 12 hours; conceivably, the run-time of an injection could end after the twelve hours.

7.5 INSTRUMENT CALIBRATION

7.5.1 Initial Calibration for Method 8270D

Prior to the analysis of samples and required method blanks, and after the instrument DFTPP tuning criteria have been met, the GC/MS system must be calibrated. The calibration consists of a six point curve. The calibration

levels are 10, 25, 50, 75, 100 and 125 ng/uL Calibration is done to determine instrument sensitivity and the linearity of GC/MS response for the semivolatile target and surrogate compounds.

Final conc. (ng/uL)	125ng /uL SVOA Stock Soln Added (uL)	1000 ug/mL dinoseb Standard (uL)	$MeCl_2$ Added (uL)	Final Vol (uL)	IS Added (uL)
10	16	2	182	200	2
25	40	5	155	200	2
50	80	10	110	200	2
75	120	15	65	200	2
100	160	20	20	200	2
125	100	0	0	100	1

If additional compound mixtures are added, the volume of $MeCl_2$ is adjusted to maintain a final volume of 200 or 100 uL. A 100 uL aliquot of each of the standards above is spiked as above with 4000 ng/uL Internal Standard stock and analyzed.

Internal Standards
1,4-Dichlorobenzene-d4
Naphthalene-d8
Acenaphthene-d10
Phenanthrene-d10
Chrysene-d12
Perylene-d12

The GC/MS operating conditions for the calibration standards injections are the same as for the DFTPP with the following exceptions:

Column Temperature Program	40°C for 3 minutes to 300°C at 10°/minute
Final Column Temperature hold	300°C
Run Time	34-36 minutes
Scan Start Time	1.8 minutes – variable, depending upon column length
Injection volume	1 uL

The conditions are set up in the method files L8270CXX.M.
After analysis of the six calibration points, they must be processed and evaluated for adherence to QC criteria. Minimum requirements of ID files are the use of specific quantitation ions and quantitating a specific set of targets and surrogates with a set internal standard. These requirements are found in Tables 3 and 5.

7.5.2 Initial Calibration Criteria

Relative response factors (RRFs) must be calculated and evaluated for each target compound and surrogate. The RRF is defined as follows:

$$RRF = \frac{Ax}{A_{IS}} \times \frac{C_{IS}}{Cx}$$

where: Ax = area of the primary ion for the target compound
A_{IS} = area of the primary ion for the corresponding istd
C_{IS} = concentration of the istd (ng/uL)
C_x = concentration of the target compound

After the calibration points have been quantitated, update the calibration curve points using the Target data processing software to generate the Mean RRF and %RSD for all analytes. If information is needed concerning the use of these programs, consult the Department Manager or a senior chemist within the group.

Response factor criteria have been established for the calibration of the semivolatile target and surrogate compounds. These criteria must be met in order for the calibration curve to be considered valid. The percent RSD for each target analyte must be less than or equal to 20%.

It is recommended that a minimum response factor (Table 6) for target analytes be achieved as a means to ensure that these compounds are behaving as expected. In addition, meeting the minimum response factor for the lowest calibration standard is critical in establishing and demonstrating the desired sensitivity. Therefore the minimum response factors in Table 6 must be verified at the lowest calibration level.

7.5.2.1 Linearity of Target Analytes (This is also applicable to clients that request DOD criteria.)

If the RSD of any target analyte is 20% or less, then the response factor is presumed to be constant over the calibration range, and the average response factor may be used for quantitation.

If the RSD of any target analyte exceeds 20%, then a calibration option outlined in section 7.0 of method 8000 will need to be employed. Please note that some options may not be allowable for certain states, federal programs, or clients. South Carolina does not allow option 2 (non-linear) for compliance work originating in their state.

Option 1 (Section 7.5.2 of method 8000 - Rev. 2, 12/96), is a linear

regression of instrument response versus the standard concentration. The correlation coefficient (r) for each target analyte and surrogate must be greater than or equal to 0.995. For linear models, Target reports r^2. This is calculated by either calculating r or squaring the result or by calculating the coefficient of determination. For a linear calibration, the equation for either is the same. The value for r^2 must greater than or equal to 0.990.

The method of linear regression analysis has the potential for a significant bias to the lower portion of a calibration curve. When calculating the calibration curves using the linear regression model, a minimum quantitation check on the viability of the lowest calibration point should be performed by re-fitting the response from the low concentration calibration standard back into the curve. The recalculated concentration of the low calibration point should be within ± 30% of the standard's true concentration. Analytes which do not meet the minimum quantitation calibration re-fitting criteria should be considered "out of control".

Corrective action such as redefining the lower limit of quantitation and/or reporting those "out of control" target analytes as estimated when the concentration is at or near the lowest calibration point may be appropriate.

Option 2 (Section 7.5.3 of method 8000 - Rev. 2, 12/96), is a non-linear calibration model not to exceed a third order (seven calibration points required) polynomial. The lab would use a quadratic model or second order polynomial. The use of a quadratic model requires six calibration points. In order for the quadratic model to be acceptable, the coefficient of determination must be greater than or equal to 0.99.

If more than 10% of the compounds in the initial calibration exceed the 20% RSD limits and do not meet the minimum correlation coefficient of determination criteria in option 1 or 2, the GCMS system is considered out of control and the calibration must be repeated. Note: Maintenance may have to be performed.

Internal standard (IS) responses and retention times in all standards must be evaluated immediately after data acquisition; if the RT for any IS changes by more than 0.50 minutes from the latest daily calibration standard, corrections must be made to the chromatographic system. If the extracted ion current profile (EICP) area for any IS changes by more than a factor of two (-50% to +100%), corrective action must be performed.

Each GC/MS system must be calibrated following system corrective

action, including ion source cleaning or repair and column removal or replacement.

If time remains in the clock after meeting the initial calibration acceptance criteria, samples may be analyzed. The calibration must be verified each twelve hour time period (time period starts from the moment of the DFTPP injection) for Method 8270. The SSTD050 in the curve may be used as the calibration verification standard as long as it meets the calibration verification acceptance criteria. All sample results must be quantitated using the initial calibration response factors.

7.5.2.2 Immediately following calibration an Independent Calibration Verification Standard must be analyzed. The percent difference for each target analyte must be less than or equal to 30%. For clients requiring DOD criteria, all project analytes must be within +/- 25% of true value.

7.5.2.3 Retention Time Windows

Retention time windows are set at the midpoint standard of the calibration curve, following every ICAL. When a CV is analyzed (and not an ICAL), the retention time windows of the daily CV must be within 30 seconds of the midpoint calibration standard of the most recent ICAL. The samples analyzed following the daily CV must have retention times within 30 seconds of those for the daily CV. Each successive daily CV must be compared to the most recent ICAL midpoint standard.

7.5.3 Continuing Calibration

A calibration verification check standard must be performed once every twelve hours immediately following analysis of the tuning compound DFTPP. This check contains all target compounds and surrogates at a concentration of 50 ng/uL.

After quantitation of the 50 ng/uL continuing calibration check, response factors must be calculated and compared to the average response factors in the initial calibration. The Target program calculates the calibration check response factors and compares them to the average RFs in the calibration curve by calculating percent differences.

- All target analytes must have a % difference of +/- 20%D in order to be considered in criteria.

- All target analytes should meet the minimum RRF criterion as in ICAL

(Table 6) in order to be considered in criteria.

These conditions must be met before method blank and/or sample analysis can begin.

The area for the internal standards in the calibration verification must be within a factor of two (-50% to 100%) from the mid-point standard of the most recent initial calibration. This is listed in the ISTD monitor report.
If the calibration verification does not meet criteria, corrective action must be taken. Depending on the situation, corrective action may be as follows:

- Re-analyze the 50 ng/uL continuing calibration check.
- Change the septum; clean the injection port; install a clean, silanized quartz liner; cut off a small portion (1" to 3") of the front end of the capillary column (this is usually performed when acid RFs are low and/or chromatography is poor).
- Analyze a new initial calibration curve.

The last option, the generation of a new initial calibration curve, is usually chosen when percent difference are >30%. In these instances, there is little or no chance of a continuing calibration reanalysis meeting criteria. If there is any doubt concerning which corrective action to undertake, consult the Department Manager or a senior chemist within the group.

If the calibration verification does meet the criteria specified above then analysis may proceed using initial calibration response factors.

7.6 SAMPLE ANALYSIS

Sample extracts may be analyzed only after the GC/MS system has met tuning criteria, initial calibration and continuing calibration requirements. Ensure that the same instrument conditions are being used for tuning, calibration and sample analysis
by reviewing the GC parameters using the “Edit entire method” option under the Method menu in MSTOP. Note that you can not edit a method if the instrument is running.

Extracts are stored in the refrigerator in the organics extraction laboratory at 4°C ±2°C. Remove them from the refrigerator and place them in the GC/MS laboratory semivolatile hood when ready for analysis.

Prepare a 1.8 mL clear glass vial (crimp top) with a disposable insert (350 uL). Add 100 uL of sample extract and 1.0 uL of the 4000 ng/uL IS stock to the vial and then cap. This gives a 40 ng/uL final concentration for the internal standard compounds.

The samples are topped with Teflon lined crimp top caps.

7.7 FINAL DATA PACKAGE

7.7.1 Initial Data Review (IDR)

The initial data review is accomplished by the analyst who analyzed the samples and is a review of sufficient quality and detail to provide a list of samples that need to be reanalyzed or diluted and reanalyzed. The initial data review is performed on the detailed quantitation reports of the analyzed sample. This data review examines criteria that directly impact whether or not the sample needs to be reanalyzed:

- Surrogate Recoveries
- Internal Standard Area Stability
- Method Blank Acceptance
- Chromatography
- Target Compound Detection/Quantitation/Review for false positives
- Laboratory Control Sample Recoveries
- Matrix Spike/Matrix Spike Duplicate Recoveries

The analyst must evaluate all data using the QA Acceptance Criteria table found within this SOP (Table 1). This table gives acceptance criteria and corrective actions for criteria that are not met. In addition to evaluating QC elements, the chromatography and quantitation of target analytes must be reviewed. During this review, the analyst checks the integration of each individual peak. The hardcopy has false positives crossed out so they can be reviewed for appropriateness by the Department Manager.

7.7.2 Chromatography

The chromatography should be examined for the presence or absence of any ghost peaks and can also be used as an indication of whether or not matrix interferences might be affecting surrogate recoveries and/or Istd area recoveries. Whether or not the chromatography is acceptable is a judgment call on the part of the analyst and should be used in conjunction with other monitored QC (e.g. surrogate recoveries) to determine the necessity of reanalyzing.

Manual integrations are to be performed when chromatographic conditions preclude the computer algorithm from correctly integrating the peak of concern. In no instance shall a manual integration be performed solely to bring a peak within criteria.

Each peak of concern is examined by the primary analyst to ensure that the peak was integrated properly by the computer algorithm. Should a manual

integration be necessary (for instance, due to a split peak, peak tailing, or incomplete resolution of isomeric pairs), an "m" qualifier will automatically be printed on the quantitation report summary. All manual integrations are initialed, dated and given a code which describes the reason for the manual integration.

This manual integration package must then be submitted to the Department Manager or his/her designee, who will review each manual integration.
For specific procedures on how to manually integrate, refer to Katahdin SOP QA-812, "Manual Integration", current revision.

7.7.3 Target Compound Detection/Quantitation
The semivolatile ID files have been set up to err on the side of false positives; that is, to identify and quantitate peaks as target compounds that may not necessarily be valid hits. It is the responsibility of the GC/MS analyst to use his/her technical judgment to determine if the identification of a target compound is correct or not.

If any target concentration exceeds the upper limit, a dilution must be made and analyzed. The dilution chosen should keep the concentration of the largest target compound hit in the upper half of the initial calibration range. LCS and MS/MSD samples need not be diluted to get spiked analytes within the calibration range.

The requirements for qualitative verification by comparison of mass spectra are as follows:

- All ions present in the standard mass spectra at a relative intensity > 10% must be present in the sample spectrum.
- The relative intensities of primary and secondary ions must agree within ±20% between the standard and sample spectra.
- Ions greater than 10% in the sample spectrum but not present in the standard spectrum must be considered and accounted for by the analyst.

If a compound cannot be verified by all three criteria above, but, in the technical judgment of the mass spectral interpretation specialist, the identification is correct, then the laboratory shall report that compound on the Form 1 as a valid hit.

The GC/MS laboratory initial data review must be completed within twelve hours of batch completion; in the majority of instances, the initial review should be accomplished at the beginning of a work shift for the previous set of analyses.

7.7.3.1 Tentatively Identified Compounds (TIC)

TIC's may be requested by certain clients for samples. Refer current Katahdin to SOP CA-207 "GC/MS Library Search and Quantitation.

7.7.4 Reporting

After the chromatograms have been reviewed and any target analytes have been quantitated using Target, the necessary files are brought into QuickForms. Depending on the QC label requested by the client, a Report of Analysis (ROA) and additional reports, such as LCS forms and chronology forms, are generated. The package is assembled to include the necessary forms and raw data. The data package is reviewed by the primary analyst and then forwarded to the secondary reviewer. The secondary reviewer validates the data and checks the package for any errors. When completed, the package is sent to the department manager for final review. A complete review checklist is provided with each package. The final data package from the Organics department is then processed by the Data Management department.

7.8 Injection Port Liner Cleaning And Silanizing Procedure

7.8.1 Remove the rubber o-ring from the liner and place the liner in a large Erlenmeyer flask.

7.8.2 In the hood, pour nitric acid into the flask until the liner is covered. Place the flask on a hotplate and boil for 2-3 hours.

7.8.3 Let cool; drain nitric acid and thoroughly flush the liner with water.

7.8.4 Bake briefly in the muffle oven until liner is dry and cool to room temperature.

7.8.5 Place the liner in a beaker, fill with Sylon and let it soak for at least two hours.

7.8.6 Take out the liner and rinse it thoroughly with toluene.

7.8.7 Rinse the liner thoroughly with purge and trap grade methanol.

7.8.8 Bake the liner in the muffle oven for a minimum of three hours.

7.9 Instrument Maintenance

Instrument preventative maintenance is performed on a semi-annual basis by GC/MS chemists. This maintenance includes a thorough inspection and cleaning of all parts, including changing rough and turbopump oils. GC/MS analysts perform other maintenance on an as-needed basis. Typically, routine maintenance involves

clipping off the front end of the DB-5MS column, replacing the injection port septum, and installing a freshly silanized quartz liner after sample analysis.

All maintenance must be documented in the instrument-specific maintenance log, whether it is routine or not. The Department Manager must authorize any maintenance over and above a routine source cleaning.

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

Refer to Table 1 and to details in this section for a summary of QC requirements, acceptance criteria, and corrective actions. These criteria are intended to be guidelines for analysts. The criteria does not cover all possible situations. If any of the QC requirements are outside the recovery ranges listed in this section or in Table 1, all associated samples must be evaluated against all the QC. In some cases data may be reported, but may be reanalyzed in other cases. Making new reagents and standards may be necessary if the standardization is suspect. The corrective actions listed in this section and in Table 1 may rely on analyst experience to make sound scientific judgements. These decisions are based on holding time considerations, client and project specific Data Quality Objectives and on review of chromatograms. The supervisor, Operations Manager, and/or Quality Assurance Officer may be consulted to evaluate data. Some samples may not be able to be reanalyzed within hold time. In these cases "qualified" data with narration may be advisable after consultation with the client.
In some cases the standard QC requirements listed in this section and in Table 1 may not be sufficient to meet the Data Quality Objectives of the specific project. Much of the work performed at the lab is analyzed in accordance with specific QC requirements spelled out in a project specific Quality Assurance Project Plan (QAPP) or in a program specific Quality Systems Manual (QSM). The reporting limits, acceptance criteria and/or corrective actions may be different than those specified in this SOP. In these cases the appropriate information will be communicated to the Department Manager and/or senior chemists before initiation of the analyses so that specific product codes can be produced for the project. In addition, the work order notes for each project will describe the specific QAPP or QSM to be followed.

8.1 Method Blank Criteria

A method blank is defined as a volume of a clean reference material (laboratory reagent grade water for water samples, baked organic-free sand for soil/sediment matrices) that is carried through the entire analytical procedure. One method blank must be extracted with each group of samples of a similar matrix and must be analyzed on the GC/MS system that was used to analyze the samples.

An acceptable method blank must contain less than or equal to the PQL of any target compound. For clients requiring DOD criteria, no analytes detected at > ½ PQL and > 1/10 the amount measured in any sample or 1/10 the regulatory limit.

If the method blank exceeds these contamination levels, the analytical system is considered out of control and corrective action must be taken before sample analysis.
Reanalysis of the blank is the first step of the corrective action; if that does not solve the problem, a Katahdin Corrective Action Report (CAR) will be initiated.

Corrective action will be specified after consultation including the Department Manager, Operations Manager, and QA Officer.

8.2 Surrogate Recoveries

There are six surrogates, which can be divided as follows:

- B/N - Nitrobenzene-d5, 2-Fluorobiphenyl and Terphenyl-d14
- Acid - Phenol-d5, 2-Fluorophenol and 2,4,6-Tribromophenol

The surrogates have laboratory derived statistical limits that are updated on an annual basis and are available in the QA office. For clients requiring DOD criteria, use acceptance limits specified by DOD or use in-house limits where none are specified.

If specifications are not met, the sample (or blank) should be reanalyzed. If specifications are met in the reanalysis, this reanalysis should only be submitted. If surrogate specifications are not met in the sample or method blank reanalysis, a Corrective Action Report (CAR) should be initiated. Corrective action will be specified after consultation including the Department Manager and Operations Manager.

For further information regarding the acceptance of surrogate recoveries, consult the Department Manager.

8.3 Internal Standard Responses

Internal standard responses and retention times (RT) in all samples and blanks must be evaluated as part of the technical data review. The method files have been set up to only detect compounds that fall within a set RT window. For Method 8270 analysis, if the extracted ion current profile (EICP) area for any internal standard changes by more than a factor of two (-50% to +100%) as compared to the daily continuing calibration standard, reanalysis must occur. If the reanalysis meets criteria, only the in-criteria run should be reported. If the reanalysis is still out-of-criteria, both analyses should be included in the sample package set.

MS/MSD samples that do not meet the EICP area criteria above do not have to be reanalyzed.

8.4 Laboratory Control Sample (LCS)

An LCS must be performed for each group of samples of a similar matrix, for the following, whichever is more frequent:

- Every 20 samples of a similar matrix or similar concentration, or
- Every batch of samples extracted.

Statistical limits are compiled annually for LCS recoveries (archived in QA office). Statistical limits are only calculated when at least 20 usable data points are obtained for any given compound. If insufficient data points are available, nominal limits are set by the Department Manager, Laboratory Operations Manager and Quality Assurance Officer. Refer to Katahdin SOP QA-808, "Generation and Implementation of Statistical QC Limits and/or Control Charts", current revision.

The use of statistical limits versus nominal limits is dependent on the client and project. This information is communicated to the Department Manager through the Katahdin project manager. It is standard practice to use statistical limits for reporting purposes and to evaluate any QC criteria exceedances. However, nominal limits of 60-140% or 70-130% may be used for some projects or states (i.e. South Carolina). For clients requiring DOD criteria, use acceptance limits specified by DOD or use in-house limits where none are specified.

The LCS recoveries for all analytes are evaluated. All of the compounds of interest must fall within the established statistical limits with the following sporadic exceedance allowances. South Carolina does not allow for marginal exceedances for compliance work originating in their state.

Number of Analytes	Number of Allowable Exceedances
> 90	5
71 – 90	4
51 – 70	3
31 – 50	2
11 – 30	1
<11	0

If less than the number of allowable exceedances fail the statistical limits, no corrective action is needed. If greater than the number of allowable exceedances fail the statistical limits, corrective action may be taken. Corrective actions may vary with each situation. However, in the case where the failures are high and the samples are non-detect for those compounds, then no corrective action is required. Otherwise, corrective action may involve reanalysis or recalibration. The specific corrective actions taken will rely on analyst experience to make sound scientific judgments while considering client objectives, other quality control indicators and/or the ability to reanalyze a sample within holding time.

Please note that for compounds with only nominal limits (i.e. insufficient data points

were available to generate statistical limits), no corrective action is required for out-of-criteria recoveries until enough data points are established to generate statistical limits.

8.5 Matrix Spike/Matrix Spike Duplicate (MS/MSD) Criteria

Matrix Spike and Matrix Spike Duplicates must be extracted and analyzed for each group of up to 20 samples of a similar matrix or similar concentration. In the event insufficient sample volume is available an LCS/LCS Duplicate is extracted and analyzed in place of the MS/MSD.

Statistical limits are compiled annually for MS/MSD recoveries for a short list of the spiked compounds. Nominal limits of 60-140% are used for all other compounds. Generally, corrective action is only taken for the short list of the spiked compounds. The specific corrective actions will rely on analyst experience to make sound scientific judgements while considering client objectives, other quality control indicators and/or the ability to reanalyze a sample within holding time. For clients requiring DOD criteria, use acceptance limits specified by DOD or use in-house limits where none are specified.

8.6 QC Requirements

Refer to Table 1 for a summary of QC requirements, acceptance criteria, and corrective actions. Table 1 criteria are intended to be guidelines for analysts. The table does not over all possible situations. If any of the QC requirements are outside the recovery ranges listed in Table 1, all associated samples must be evaluated against all of the QC. In some cases data may be reported, but may be reanalyzed in other cases. Making new reagents and standards may be necessary if the standardization is suspect. The corrective actions listed in Table 1 may rely on analyst experience to make sound scientific judgments. These decisions are based on holding time considerations, client and project specific Data Quality Objectives and on review of chromatograms. The Department Manager, Operations Manager, and/or Quality Assurance Officer may be consulted to evaluate data. Samples may not be able to be reanalyzed within hold time. In these cases "qualified" data with narration may be advisable after consultation with the client.

9.0 METHOD PERFORMANCE

The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero. The MDLs are determined annually per type of instrument and filed with the Organic Department Manager and with the QAO. Refer to the current revision of Katahdin SOP QA-806, Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications, for procedures on determining the MDL.

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Refer to the current revision of Method 8270 for other method performance parameters and requirements.

10.0 APPLICABLE DOCUMENTS/REFERENCES

Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, USEPA SW846, 3rd Edition, Final Updates I, II, IIA, IIB, III, IIIA, IIIB and IV, February 2007, Method 8270D.

Katahdin SOP CA-101, Equipment Maintenance, current revision.

Department of Defense Quality Systems Manual for Environmental Laboratories (DoD QSM), Version 4.1, 04/22/09

The National Environmental Laboratory Accreditation Conference (NELAC) Standards, June 2003

LIST OF TABLES AND FIGURES

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY CAPILLARY COLUMN GC/MS: SW 846 METHOD 8270D.

TABLE 1

QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Check of mass spectral ion intensities using DFTPP	Prior to initial calibration and calibration verification	Refer to the criteria listed in Section 7.4	Retune instrument, and verify
Six-point initial calibration for all analytes	Initial calibration prior to sample analysis	RSD 20% for all compounds. If not met: Option 1) Linear least squares regression: $r \geq 0.995$ Option 2) Non-linear regression: coefficient of determination (COD) $r^2 \geq 0.99$ (6 points for second order) Up to 10% target analytes may be outside of the above criteria Refer to section 7.5.2.1 for additional information.	Perform instrument maintenance if necessary. Repeat calibration if criterion is not met
Independent calibration verification	Once after Initial calibration	± 30 % D	1) Reanalyze standard 2) Reprep standard 3) Reprep standard from fresh stock.
Continuing calibration verification	Once per each 12 hours, prior to sample analysis	All target analytes: $\leq$ 20%D	Repeat initial calibration and reanalyze all samples analyzed since the last successful calibration verification
ISs	Immediately after or during data acquisition of calibration check standard	Retention time $\pm$ 30 seconds; EICP area within -50% to +100% of last calibration verification (12 hours) for each IS	Inspect mass spectrometer or GC for malfunctions; mandatory reanalysis of samples analyzed while system was malfunctioning
Demonstration of ability to generate acceptable accuracy and precision	Once per analyst and annually there after.	All recoveries within method QC acceptance limits.	Recalculate results; locate and fix problem; reextract/reanalyze P&A study for those analytes that did not meet criteria
Method blank	One per prep batch	No analytes detected > PQL	1) Investigate source of contamination 2) Evaluate the samples and associated QC: i.e.If the blank results are above the PQL, report samples that are <PQL or > 10X the blank result. Reprep a blank and the remaining samples.

TABLE 1, (cont.)

QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
LCS for all analytes	One LCS per prep batch	Statistically derived from lab data or nominal limits depending on the project. Refer to QA records for statistical limits. Nominal limits are used as default limits. See also section 8.4 of this SOP for more information on allowable exceedances.	1) Evaluate the samples and associated QC: i.e.If an MS/MSD was performed and acceptable, narrate. 2) If an LCS/LCSD was performed and only one was unacceptable, narrate. 3) If the surrogate recoveries in the LCS are low but are acceptable in the blank and samples, narrate. 4) If the LCS rec. is high but the sample results are <PQL, narrate. 5) Otherwise, reprep a blank and the remaining samples.
Surrogate spike	Every sample, control, standard, and method blank	Current statistical limits	1) Check chromatogram for interference; if found, flag data 2) If not found, check instrument performance; if problem is found, correct and reanalyze 3) If still out reextract and analyze sample 4) (4) If reanalysis is out, flag data
MS/MSD	One MS/MSD per every 20 samples	Statistically derived from lab data or nominal limits depending on the project. Refer to QA records for statistical limits and section 8.5 of this SOP.	1) Evaluate the samples and associated QC: i.e. If the LCS results are acceptable, narrate. 2) (2) If both the LCS and MS/MSD are unacceptable reprep the samples and QC.
MDL study	Refer to KAS SOP QA-806, “Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications”, current revision.		

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TABLE 2

SUMMARY OF METHOD MODIFICATIONS

Topic	Katahdin SOP CA-226-01	Method 8270, current revision
Apparatus/Materials	none	
Reagents	none	
Sample preservation/ handling	none	
Procedures	none	
QC - Spikes	none	
QC - LCS	none	
QC - Accuracy/Precision	none	
QC - MDL	PQL – Practical Quantitation Level – three to ten times the MDL.	EQL – Estimated Quantitation Level – five to ten times the MDL

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TABLE 3

Analyte Quantitation and Internal Standards

Internal Standard: 1,4-dichlorobenzene-d4

Target and Surrogates:

Pyridine (not on TCL list)
N-Nitrosodimethylamine (not on TCL list)
Aniline (not on TCL list)
Phenol
Bis (2-chloroethyl) ether
2-Chlorophenol
1,3-Dichlorobenzene
1,4-Dichlorobenzene
1,2-Dichlorobenzene
Benzyl alcohol (not on PP list)
2-Methylphenol (not on PP list)
2,2'-oxybis(1-chloropropane) (also known as Bis (2-Chloroisopropyl) ether)
4-Methylphenol (not on PP list)
N-Nitroso-di-n-propylamine
Hexachloroethane
Ethyl methanesulfonate (8270 C)
Methyl methanesulfonate (8270 C)
2-Picoline (8270 C)
N-Nitrosomethylethylamine (Appendix IX)
N-Nitrosodiethylamine (Appendix IX)
N-Nitrosopyrrolidine (Appendix IX)
N-Nitrosomorpholine (Appendix IX)
2-Fluorophenol (surrogate)
Phenol-d6 (surrogate)

Internal Standard: Naphthalene-d8

Target and Surrogates:

Nitrobenzene
Isophorone
2-Nitrophenol
2,4-Dimethylphenol
Acetophenone (8270 C)
Benzoic acid (not on PP list)
Bis (2-chloroethoxy) methane
2,4-Dichlorophenol
2,6-Dichlorophenol (8270 C)
1,2,4-Trichlorobenzene
a, a-Dimethyl-phenethylamine (8270 C)
Naphthalene
4-Chloroaniline (not on PP list)
Hexachlorobutadiene
4-Chloro-3-methylphenol
2-Methylnaphthalene
N-Nitrosodi-n-butylamine (8270 C)
N-Nitrosopiperidine (8270 C)
o-toluidine (Appendix IX)
o, o, o-Triethylphosphorothioate (Appendix IX)
Hexachloropropene (Appendix IX)
Isosafrole (Appendix IX)
Nitrobenzene-d5 (surrogate)

Internal Standard: Acenaphthene-d10

Target and Surrogates:

Hexachlorocyclopentadiene
2,4,6-Trichlorophenol
2,4,5-Trichlorophenol (not on PP list)
1-Chloronaphthalene (8270 C)
2-Chloronaphthalene
2-Nitroaniline (not on PP list)
Dimethyl phthalate
Acenaphthylene
3-Nitroaniline (not on PP list)
Acenaphthene
2,4-Dinitrophenol
4-Nitrophenol
Dibenzofuran (not on PP list)
2,4-Dinitrotoluene
2,6-Dinitrotoluene
Diethyl phthalate
4-Chlorophenylphenyl ether
Fluorene
4-Nitroaniline (not on PP list)
1-Naphthylamine (8270 C)
2-Naphthylamine (8270 C)
Pentachlorobenzene (8270 C)

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY CAPILLARY COLUMN GC/MS: SW 846 METHOD 8270D.

TABLE 3 (cont.)

Analyte Quantitation and Internal Standards

1, 2, 4, 5-Tetrachlorobenzene (8270 C)
2, 3, 4, 6-Tetrachlorophenol (8270 C)
p-Phenylenediamene (Appendix IX)
Safrole (Appendix IX)
1,4-Naphthoquinone (Appendix IX)
Thionazine (Appendix IX)
5-Nitro-o-toluidine (Appendix IX)
1,2-Diphenylhydrazine (not on TCL list)
2-Fluorobiphenyl (surrogate)
2,4,6-Tribromophenol (surrogate)

Internal Standard: Phenanthrene-d10

Target and Surrogates:

4,6-Dinitro-2-methylphenol
N-Nitrosodiphenylamine
Diphenylamine (8270 C)
4-Bromophenylphenyl ether
Phenacetin (8270 C)
Hexachlorobenzene
4-Aminobiphenyl (8270 C)
Pentachlorophenol
Pentachloronitrobenzene (8270 C)
Pronamide (8270 C)
Phenanthrene
Anthracene
Di-n-butylphthalate
Carbazole (8270 B)
Fluoranthene
Sym-Trinitrobenzene (Appendix IX)
Diallate (Appendix IX)
4-Nitroquinoline-1-oxide (Appendix IX)
Methapyrilene (Appendix IX)
Isodrin (Appendix IX)

Internal Standard: Chrysene-d12

Target and Surrogates:

Benzidine (not on TCL list)
Pyrene
Butylbenzyl phthalate
3,3'-Dichlorobenzidine
p-Dimethylaminoazobenzene (8270 C)
Benzo (a) Anthracene
Bis (2-ethylhexyl) phthalate
Chrysene
3-Methylcholanthrene (8270 C)
Aramite (Appendix IX)
Chlorobenzilate (Appendix IX)
3,3'-Dimethylbenzidine (Appendix IX)
2-Acetylaminofluorene (Appendix IX)
Terphenyl-d14 (surrogate)

Internal Standard: Perylene-d12

Target and Surrogates:

Di-n-octyl phthalate
Benzo (b) fluoranthene
Benzo (k) fluoranthene
Benzo (a) pyrene
Indeno (1,2,3-cd) pyrene
Dibenz (a, h) anthracene
Dibenz (a, j) acridine (8270 C)
Benzo (ghi) perylene
7,12-Dimethylbenz (a) anthracene (8270 C)
Hexachlorophene (Appendix IX)

TABLE 4

PROCEDURE CONDENSATION

Clock

12 hours from injection of 50ng DFTPP.

Calibration Curve Criteria

RSD 20% for all compounds.
If not met:
Option 1) Linear least squares regression: $r \geq 0.995$
Option 2) Non-linear regression: coefficient of determination (COD) $r^2 \geq 0.99$ (6 points for second order)
Up to 10% of target analytes may be outside of the above criteria
Refer to section 7.5.2.1 for additional information.
Recommended minimum RF criteria for analytes listed in Table 6.

Continuing Calibration Check Criteria

All target analytes: $\leq$ 20%D
Recommended minimum RF criteria for analytes listed in Table 6.

Additional QC

LCS every extraction batch
MS/MSD every 20 samples

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY CAPILLARY COLUMN GC/MS: SW 846 METHOD 8270D.

TABLE 5

CHARACTERISTIC IONS FOR SEMIVOLATILE COMPOUNDS

COMPOUND	PRIMARY ION	SECONDARY ION(S)
2-Picoline	93	66,92
Aniline	93	66,65
N-Nitrosodimethylamine	42	74,43
Phenol	94	65,66
Bis(2-Chloroethyl)ether	93	63,95
2-Chlorophenol	128	64,130
1,3-Dichlorobenzene	146	148,111
1,4-Dichlorobenzene	146	148,111
1,2-Dichlorobenzene	146	148,111
N-Nitrosomethylethylamine	88	42,43,56
Benzyl alcohol	108	77,79
2-Methylphenol	107	107,108,77,79,90
Bis(2-Chloroisopropyl)ether	45	77,121
4-Methylphenol	107	107,108,77,79,90
N-Nitroso-di-n-propylamine	70	42,101,130
Hexachloroethane	117	201,199
Nitrobenzene	77	123,65
Isophorone	82	95,138
2-Nitrophenol	139	65,109
2,4-Dimethylphenol	122	121,107
Benzoic acid	122	105,77
Bis(2-chloroethoxy)methane	93	95,123
2,4-Dichlorophenol	162	164,98
1,2,4-Trichlorobenzene	180	182,145
Naphthalene	128	129,127
4-Chloroaniline	127	129,65,92
Hexachlorobutadiene	225	223,227
4-Chloro-3-methylphenol	107	144,142
2-Methylnaphthalene	142	141
Hexachlorocyclopentadiene	237	235,272
2,4,6-Trichlorophenol	196	198,200
2,4,5-Trichlorophenol	196	198,97,132,99
2-Chloronaphthalene	162	164,127
2-Nitroaniline	65	92,138
Dimethyl phthalate	163	194,164
Acenaphthylene	152	151,153
3-Nitroaniline	138	108,92
Acenaphthene	153	152,154
2,4-Dinitrophenol	184	63,154
4-Nitrophenol	109	139,65
Dibenzofuran	168	139
2,4-Dinitrotoluene	165	63,89
2,6-Dinitrotoluene	165	89,63
Diethyl phthalate	149	177,150
4-Chlorophenylphenylether	204	206,141
Fluorene	166	165,167
4-Nitroaniline	138	92,108,65,80,39
4,6-Dinitro-2-methylphenol	198	105,51
N-Nitrosodiphenylamine	169	168,167
4-Bromophenylphenylether	248	250,141

TABLE 5 (cont.)

CHARACTERISTIC IONS FOR SEMIVOLATILE COMPOUNDS

COMPOUND	PRIMARY ION	SECONDARY ION(S)
Hexachlorobenzene	284	142,249
1,2-Diphenylhydrazine	184	77,92
Pentachlorophenol	266	264,268
Phenanthrene	178	179,176
Di-n-butyl phthalate	149	150,104
Carbazole	167	166,139
Fluoranthene	202	101,203
Benzidine	184	92,185
Pyrene	202	200,203
Butylbenzylphthalate	149	91,206
3,3-Dichlorobenzidine	252	254,126
Benzo(a)anthracene	228	229,226
Bis(2-ethylhexyl)phthalate	149	167,279
Chrysene	228	229,226
Di-n-octyl phthalate	149	167,43
Benzo(b)fluoranthene	252	253,125
Benzo(k)fluoranthene	252	253,125
Benzo(a)pyrene	252	253,125
Indeno(1,2,3-cd)pyrene	276	138,277
Dibenz(ah)anthracene	278	139,279
Benzo(ghi)perylene	276	138,277
N-Nitrosodiethylamine	102	42,57,44,56
N-Nitrosopyrrolidine	100	41,42,68,69
N-Nitrosomorpholine	56	116,86
Acetophenone	105	71,51,120
2,6-Dichlorophenol	162	63,98
α,α-Dimethylphenethylamine	58	91,65,134,42
N-Nitrosodi-n-butylamine	84	57,41,116,158
N-Nitrosopiperidine	114	42,55,56,41
O-toluidine	106	107,77,51,79
O,O,O-Triethylphosphorothioate	198	121,97,65
Hexachloropropene	213	211,215,117,106,141
Isosafrole	162	131,104,77,51
1-Chloronaphthalene	162	127,164
1-Naphthylamine	143	115,89,63
2-Naphthylamine	143	115,116
Pentachlorobenzene	250	252,108,248,215,254
1,2,4,5-Tetrachlorobenzene	216	214,179,108,143,218
2,3,4,6-Tetrachlorophenol	232	131,230,166,234,168
p-Phenylenediamene	108	80,53,54,52
Safrole	162	104,77,103,135
1,4-Naphthquinone	158	104,102,76,50,130
Thionazine	107	96,97,143,79,68
5-Nitro-o-toluidine	152	77,79,106,94
4-Aminobiphenyl	169	168,170,115
Diphenylamine	169	168,167
Pentachloronitrobenzene	237	142,214,249,295,265
Phenacetin	108	180,179,109,137,80
Pronamide	173	175,145,109,147
sym-Trinitrobenzene	75	213,120

TABLE 5 (cont.)

CHARACTERISTIC IONS FOR SEMIVOLATILE COMPOUNDS

COMPOUND	PRIMARY ION	SECONDARY ION(S)
Diallate	86	234,43,70
4-Nitroquinoline-1-oxide	174	101,128,75,116
Methapyrilene	97	50,191,71
Isodrin	193	66,195,263,265,147
p-Dimethylaminoazobenzene	225	120,77,105,148,42
7,12-Dimethylbenz(a)anthracene	256	241,239,120
3-Methylcholanthrene	268	252,253,126,134,113
Aramite	185	191,319,334,197,321
Chlorobenzilate	251	139,253,111,141
3,3'-Dimethylbenzidine	212	106,196,180
2-Acetylaminofluorene	181	180,223,152
Dibenz(a,j)acridine	279	280,277,250
Hexachlorophene	196	198,209,21,406,408
Phenol-d6 (surrogate)	99	42,71
2-Fluorophenol (surrogate)	112	64
2,4,6-Tribromophenol (surrogate)	330	332,141
Nitrobenzene-d5 (surrogate)	82	128,54
2-Fluorobiphenyl (surrogate)	172	171
Terphenyl-d14 (surrogate)	244	122,212
1,4-Dichlorobenzene-d4 (istd.)	152	115,150
Naphthalene-d8 (istd.)	136	68
Acenaphthene-d10 (istd.)	164	162,160
Phenanthrene-d10 (istd.)	188	94,80
Chrysene-d12 (istd.)	240	120,236
Perylene-d12 (istd.)	264	260,265

Primary ions must not be changed except in unusual instances where interference occurs with a co-eluting non-target analyte. In this case, a secondary ion may be used for quantitation with the following rules:

(1) The corresponding standard(s) (initial calibration curve and continuing calibration standard) must be re-quantitated with the secondary ion.

(2) Approval must be obtained from the Department Manager or the laboratory Operations Manager.

The quantitation ion must then be changed back to the one specified in Table 3 after quantitation of the samples(s).

Secondary ions are recommended only and may be changed depending upon instrument conditions (sensitivity, etc.). However, it is Katahdin policy that a minimum of 2 ions (primary and one secondary) be used for all GC/MS analyses.

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY CAPILLARY COLUMN GC/MS: SW 846 METHOD 8270D.

Table 6

RECOMMENDED MINIMUM RESPONSE FACTOR FOR INITIAL AND CONTINUING CALIBRATION

Semivolatile Compounds	Minimum Response Factor (RF)
Benzaldehyde	0.010
Phenol	0.800
Bis(2-chloroethyl)ether	0.700
2-Chlorophenol	0.800
2-Methylphenol	0.700
2,2'-Oxybis-(1-chloropropane)	0.010
Acetophenone	0.010
4-Methylphenol	0.600
N-Nitroso-di-n-propylamine	0.500
Hexachloroethane	0.300
Nitrobenzene	0.200
Isophorone	0.400
2-Nitrophenol	0.100
2,4-Dimethylphenol	0.200
Bis(2-chloroethoxy)methane	0.300
2,4-Dichlorophenol	0.200
Naphthalene	0.700
4-Chloroaniline	0.010
Hexachlorobutadiene	0.010
Caprolactam	0.010
4-Chloro-3-methylphenol	0.200
2-Methylnaphthalene	0.400
Hexachlorocyclopentadiene	0.050
2,4,6-Trichlorophenol	0.200
2,4,5-Trichlorophenol	0.200
1,1'-Biphenyl	0.010
2-Chloronaphthalene	0.800
2-Nitroaniline	0.010
Dimethyl phthalate	0.010
2,6-Dinitrotoluene	0.200
Acenaphthylene	0.900
3-Nitroaniline	0.010
Acenaphthene	0.900
2,4-Dinitrophenol	0.010
4-Nitrophenol	0.010
Dibenzofuran	0.800
2,4-Dinitrotoluene	0.200
Diethyl phthalate	0.010
1,2,4,5-Tetrachlorobenzene	0.010
4-Chlorophenyl-phenyl ether	0.400
Fluorene	0.900
4-Nitroaniline	0.010
4,6-Dinitro-2-methylphenol	0.010
4-Bromophenyl-phenyl ether	0.100
N-Nitrosodiphenylamine	0.010
Hexachlorobenzene	0.100

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY CAPILLARY COLUMN GC/MS: SW 846 METHOD 8270D.

Table 6

RECOMMENDED MINIMUM RESPONSE FACTOR FOR INITIAL AND CONTINUING CALIBRATION (CONT.)

Semivolatile Compounds	Minimum Response Factor (RF)
Atrazine	0.010
Pentachlorophenol	0.050
Phenanthrene	0.700
Anthracene	0.700
Carbazole	0.010
Di-n-butyl phthalate	0.010
Fluoranthene	0.600
Pyrene	0.600
Butyl benzyl phthalate	0.010
3,3'-Dichlorobenzidine	0.010
Benzo(a)anthracene	0.800
Chrysene	0.700
Bis-(2-ethylhexyl)phthalate	0.010
Di-n-octyl phthalate	0.010
Benzo(b)fluoranthene	0.700
Benzo(k)fluoranthene	0.700
Benzo(a)pyrene	0.700
Indeno(1,2,3-cd)pyrene	0.500
Dibenz(a,h)anthracene	0.400
Benzo(g,h,i)perylene	0.500
2,3,4,6-Tetrachlorophenol	0.010

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY CAPILLARY COLUMN GC/MS: SW 846 METHOD 8270D.

FIGURE 1

EXAMPLE OF RUNLOG LOGBOOK PAGE

KATAHDIN ANALYTICAL SERVICES
GC/MS SVOA INJ LOG INSTRUMENT: 5973-U

DATE OF DFTPP INJECTION: 010708

JOB	SAMPLE	DATAFILE	DF	ALS #	METHOD	UL INJ	CHEMIST	COMMENTS
	50 ng DFTPP	UD626	1	1	DFTPP3Q0	2.0	JW	OK
B	SSTD050UO107	UO563		2	US270C02	1.0		✓
	010	64		3				✓
	025	65		4				✓ Qunne OK
	100	66		5				✓
	150	67		6				✓
	200	68		7				✓
A	SSTD 030 UO107	69(a)		8				✓ (a) = UTCLP02
	010	70(a)		9				✓ files
	025	71(a)		10				✓
	100	72(a)		11				✓
	150	73(a)		12				✓
	200	74(a)		13				✓
	8270 IND CHK	75(a)		14				

JW 010808

STANDARD	CODE
DFTPP	51176
CAL. STD.	51181 51182
IS MIX	STK 3505

REVIEWED AND APPROVED BY: _____
DATE: _____

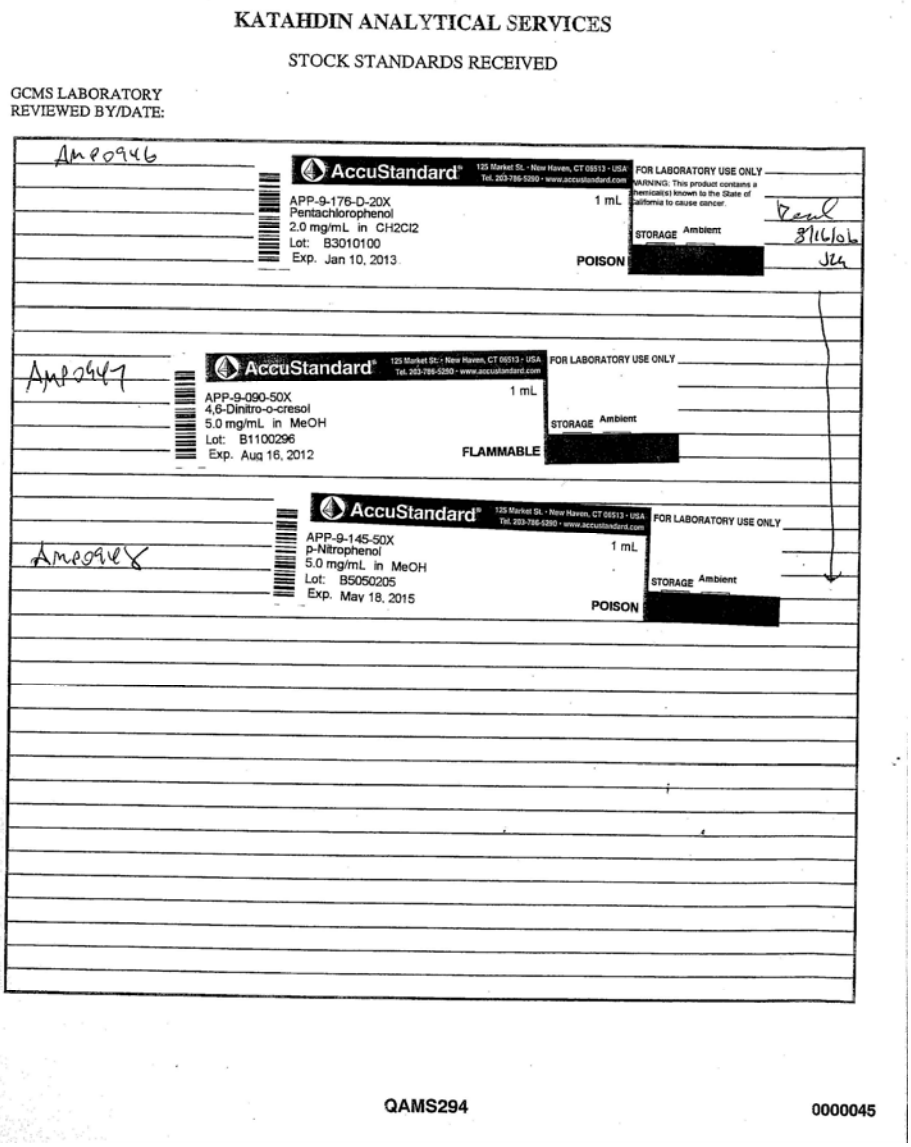
QAMS364

0000015

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY CAPILLARY COLUMN GC/MS: SW 846 METHOD 8270D.

FIGURE 2

EXAMPLE OF GC/MS STANDARDS RECEIPT LOGBOOK ENTRY



KATAHDIN ANALYTICAL SERVICES

STOCK STANDARDS RECEIVED

GCMS LABORATORY
REVIEWED BY/DATE:

AMP0946

AccuStandard® 125 Market St. · New Haven, CT 06513 · USA Tel. 203-786-5290 · www.accustandard.com

APP-9-176-D-20X
Pentachlorophenol
2.0 mg/mL in CH2Cl2
Lot: B3010100
Exp. Jan 10, 2013

1 mL

FOR LABORATORY USE ONLY
WARNING: This product contains a chemical(s) known to the State of California to cause cancer.

STORAGE Ambient

POISON [redacted]

Dewl 3/16/06 JLG

AMP0947

AccuStandard® 125 Market St. · New Haven, CT 06513 · USA Tel. 203-786-5290 · www.accustandard.com

APP-9-090-50X
4,6-Dinitro-o-cresol
5.0 mg/mL in MeOH
Lot: B1100296
Exp. Aug 16, 2012

1 mL

FOR LABORATORY USE ONLY

STORAGE Ambient

FLAMMABLE [redacted]

AMP0948

AccuStandard® 125 Market St. · New Haven, CT 06513 · USA Tel. 203-786-5290 · www.accustandard.com

APP-9-145-50X
P-Nitrophenol
5.0 mg/mL in MeOH
Lot: B5050205
Exp. May 18, 2015

1 mL

FOR LABORATORY USE ONLY

STORAGE Ambient

POISON [redacted]

QAMS294 0000045

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY CAPILLARY COLUMN GC/MS: SW 846 METHOD 8270D.

FIGURE 3

EXAMPLE OF SVOA STANDARDS PREPARATION LOGBOOK ENTRY

GC/MS SVOA STANDARD PREP LOGBOOK

50863	8270 Stock (w/o MeOH)	3-15-06	7-7-06	JLK	AMP0844	8270 Megamix	300	2-22-07	4.2ml	150 ug/ml
					AMP0937	↓	350	3-15-07		
					AMP0911	APP IX #2	600	3-2-07		
					AMP0910	↓ 1	180	3-9-07		
					AMP0570	↓ 1	200	7-7-06		
					AMP0699	Organophosphat	300	8-14-06		
					AMP0931	Benzoic Acid	↓	3-9-07		
					AMP0907	Hexachlorophene		2-22-07		
					AMP0806	Benzidine		3-9-07		
					AMP0936	3,3'-Dichlorobenzidine		3-14-07		
					AMP0932	8270 Surr	150	3-9-07		
					50861	DCA	300	3-13-07		
					BY3E90	MeCl2	550	~		
50864	8270 level 1	3-15-06	7-7-06	JLK	50863	8270 Stock	70	7-7-06	1.05ml	10 ug/ml
					BY3E90	MeCl2	980			
50865	8270 level 2	3-15-06	7-7-06	JLK	50863	8270 Stock	150	7-7-06	0.90ml	25 ug/ml
					BY3E90	MeCl2	750			
50866	8270 level 3	3-15-06	7-7-06	JLK	50863	8270 Stock	600	7-7-06	1.8ml	50 ug/ml
					BY3E90	MeCl2	1200			
50867	8270 level 4	3-15-06	7-7-06	JLK	50864	8270 Stock	700	7-7-06	1.05ml	100 ug/ml
					BY3E90	MeCl2	350			

Reviewed by/Date:

CAMS284

0000057

TITLE: ANALYSIS OF PCBs AS TOTAL AROCLORS BY GAS CHROMATOGRAPHY/ ELECTRON CAPTURE DETECTOR (GC/ECD): SW-846 METHOD 8082

Prepared By: Peter Lemay Date: 4/98

Approved By:

Group Supervisor: Peter Lemay Date: 1/15/01

Operations Manager: John C. Bunton Date: 1/15/01

QA Officer: Deborah J. Nadeau Date: 1.22.01

General Manager: Duncan C. Kilgore Date: 1/16/01

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
01 8082	Format changes, added pollution prevention, minor changes to sections 7, 8 and Table 1	DN	1.22.01	1/22/01
02 8082	Revised sections 7.3.1, 7.4.5 and 7.6.1 to be compliant with South Carolina requirements.	DN	5.23.01	5.23.01
03 8082	Changed to practice of reporting higher value. Other minor changes to sections 7.5.2, 7.7.3 + to Table 2.	DN	5.21.02	5.21.02
04 8082	Revised SOP to indicate Turbochrom is being used as instrument control + data collection software. Included Target-related definitions. Changes to sections 7.7.3, 7.7.4 and 7.8.	MRC	08.20.04	08.20.04
05 8082	Changed 7.5.2 to reflect alternating CV Changed Table 2 Sect. 7.3.1 New Checklist added wording to sect. 8	LAD	020305	020305

TITLE: ANALYSIS OF PCBs AS TOTAL AROCLORS BY GAS CHROMATOGRAPHY/ ELECTRON CAPTURE DETECTOR (GC/ECD): SW-846 METHOD 8082

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
06 8082	Changed PCB1260 to Aroclor 1260. Removed references to 3541. Updated table 2. Added instructions to shake extract before vialing	LAD	04/06	04/06
07	Added waste streams to sect. 1.0. Added ICV to definitions, sect. 5, sect. 7 and Table 1. Added wording regarding 2nd column confirmation criteria and flagging rules to sect. 7.7.4. Added CCV criteria to sect. 7.5.3 and Table 1. Added wording regarding MI to sect. 7.7.3	LAD	08/07	08/07
08	Added tissue, wipe and oil matrices. Added extraction method 3535. Added DDT analog interference, Std. information and analysis frequency criteria. Added HTs are a recommendation. Added note that 2 detectors must be used for dual column. Updated method references Removed calibration and surrogate method mod. from Table 2. Added more info @ linear calib. Added extraction references,	LAD	02/09	02/09
09	Added Chemstation to definitions. Clarified that surrogates are added to only the aroclor 1660 standards, not ALL standards.	LAD	05/09	05/09
10	Revised sections 7, 8, and 10 to reflect compliance with the DoD QSM version 4.1	LAD	08/09	08/09
11	Added Table 2 with DoD QSM Ver. 4.1 QC criteria. Minor changes to Table 1.	LAD	04/10	04/10

TITLE: ANALYSIS OF PCBs AS TOTAL AROCLORS BY GAS CHROMATOGRAPHY/ ELECTRON CAPTURE DETECTOR (GC/ECD): SW-846 METHOD 8082

Please acknowledge receipt of this standard operating procedure by signing and dating both of the spaces provided. Return the bottom half of this sheet to the QA Department.

I acknowledge receipt of copy ___ of document **SOP CA-329-11**, titled **ANALYSIS OF AS TOTAL AROCLORS BY GAS CHROMATOGRAPHY/ELECTRON CAPTURE DETECTOR (GC/ECD): SW-846 METHOD 8082**.

Recipient: _____ Date: _____

KATAHDIN ANALYTICAL SERVICES, INC.
STANDARD OPERATING PROCEDURE

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Recipient: _____ Date: _____

TITLE: ANALYSIS OF PCBs AS TOTAL AROCLORS BY GAS CHROMATOGRAPHY/ ELECTRON CAPTURE DETECTOR (GC/ECD): SW-846 METHOD 8082

1.0 SCOPE AND APPLICATION

This SOP describes all aspects of the analysis of extracts of aqueous, solid, tissue, wipe and oil samples for PCBs by EPA Method 8082A as performed by Katahdin Analytical Services, Inc. including sample analysis, data review, standard preparation and instrument calibration.

It is applicable to the following compounds: Aroclor-1242, Aroclor-1254, Aroclor-1221, Aroclor-1232, Aroclor-1248, Aroclor-1260 and Aroclor-1016. Extracts are analyzed by Gas Chromatography-Electron Capture Detector (GC-ECD)

1.1 Definitions

ANALYTICAL BATCH: 20 or fewer samples which are analyzed together with the same method sequence and the same lots of reagents and with the manipulations common to each sample within the same time period or in continuous sequential time periods.

METHOD BLANK (laboratory reagent blank): An artificial sample designed to determine if method analytes or other interferences are present in the laboratory environment, the reagents, or the apparatus. For aqueous samples, laboratory reagent grade water is used as a blank matrix; for soil samples, muffled sand is used as a blank matrix. The blank is taken through the appropriate steps of the process.

CALIBRATION CHECK: Verification of the ratio of instrument response to analyte amount, a calibration check is done by analyzing for analyte standards in an appropriate solvent. Calibration check solutions are made from a stock solution that is different from the stock used to prepare standards.

CALIBRATION STANDARD (WORKING STANDARD): A solution prepared from the stock standard solution that is used to calibrate the instrument response with respect to analyte concentration.

INDEPENDENT CALIBRATION VERIFICATION STANDARD (ICV): A solution prepared from a stock standard solution independent of the calibration mix that is used to verify the calibration.

LABORATORY CONTROL SAMPLE (LCS): A blank that has been spiked with the analyte(s) from an independent source and is analyzed exactly like a sample. Its purpose is to determine whether the methodology is in control, and whether the laboratory is capable of making accurate and precise measurements. The matrix used should be phase matched with the samples and well characterized.

MATRIX SPIKE/MATRIX SPIKE DUPLICATE (MS/MSD): Predetermined quantities of stock solutions of certain analytes are added to a sample matrix prior to sample extraction and analysis. Samples are split into duplicates, spiked and analyzed. Percent

recoveries are calculated for each of the analytes detected. The relative percent difference between the samples is calculated and used to assess analytical precision.

STANDARD CURVE (CALIBRATION CURVE): A curve that plots concentration of known analyte standard versus the instrument response to the analyte.

STOCK STANDARD SOLUTION: A concentrated solution containing a single certified standard that is a method analyte, or a concentrated solution of a single analyte prepared in the laboratory with an assay reference compound. Stock standard solutions are used to prepare calibration standards.

SURROGATES: Organic compounds which are similar to analytes of interest in chemical composition, extraction and chromatography, but which are not normally found in environmental samples. These compounds are spiked into all blanks, Aroclor 1660 standard, samples and spiked samples prior to analysis. Percent recoveries are calculated for each surrogate.

KATAHDIN INFORMATION MANAGEMENT SYSTEM (KIMS): A complete multi-user system with the capabilities of integrating laboratory instrumentation, generating laboratory worksheets, providing complete Lab Order status and generating reports. KIMS utilizes these features through a database.

PE NELSON TURBOCHROM OR HP CHEMSTATION: data acquisition systems that are used to collect chromatographic data. The systems can also be used to archive raw data files.

TARGET: A software system that combines full processing, reporting and comprehensive review capabilities, regardless of chromatographic vendor and data type.

TARGET DB: An oracle database used to store and organize all Target data files.

QUICKFORMS: A laboratory reporting software for Target and Target DB. The QuickForms report module for Target is preconfigured with generalized forms and US EPA CLP report forms and disk deliverables, which can be customized.

1.2 Responsibilities

1.2.1 This method is restricted to use by, or under the supervision of analysts experienced in the analysis of PCBs by method 8082. Each analyst must demonstrate and document their ability to generate acceptable results with this method. Refer to Katahdin SOP QA-805, “Personnel Training & Documentation of Capability,” current revision.

1.2.2 It is the responsibility of all Katahdin technical personnel involved in analysis by method 8082 to read and understand this SOP, adhere to the procedures outlined, and to properly document their data in the appropriate lab notebook. Any deviations from the test or irregularities with the samples should also be recorded in the lab notebook and reported to the Department Manager or designated qualified data reviewer responsible for this data.

1.2.3 It is the responsibility of the Department Manager to oversee that members of their group follow this SOP, to ensure that their work is properly documented and to initiate periodic review of the associated logbooks.

1.3 Health and Safety

1.3.1 Users of this procedure must be cognizant of inherent laboratory hazards, proper disposal procedures for contaminated materials and appropriate segregation of hazardous wastes. The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical should be treated as a potential health hazard. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Everyone involved with the procedure must be familiar with the MSDSs (material safety data sheets) for all the materials used in this procedure.

1.3.2 Each qualified analyst or technician must be familiar with Katahdin Analytical Environmental Health and Safety Manual including the Katahdin Hazardous Waste Plan and must follow appropriate procedures. These include the use of appropriate personal protective equipment (PPE) such as safety glasses, gloves and lab coats when working with chemicals or near an instrument and not taking food or drink into the laboratory. Each analyst should know the location of all safety equipment. Each analyst shall receive a safety orientation from their Department Manager, or designee, appropriate for the job functions they will perform.

1.4 Pollution Prevention/Waste Disposal

1.4.1 Whenever possible, laboratory personnel should use pollution prevention techniques to address their waste generation. Refer to the current revision of the Katahdin Hazardous Management Program for further details on pollution prevention techniques.

1.4.2 Wastes generated during the preparation of samples must be disposed of in accordance with the Katahdin Analytical Environmental Health and Safety Manual and SOP SD-903, “Sample Disposal,” current revision. Expired standards are lab packed, placed in the Katahdin hazardous waste storage area, and disposed of in accordance with this SOP.

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1.4.3 Wastes generated during standards preparation are disposed of in the Mixed Flammable Waste (O). After the extracts have been analyzed, the autosampler vials and any expired standard vials or ampules are disposed of in the PCB Vial Waste (H).

2.0 SUMMARY OF METHOD

2.1 Method 8082 provides gas chromatographic conditions for the detection of PPB concentrations of certain PCBs. Prior to the use of this method, appropriate sample extraction techniques must be used. Both neat and diluted organic liquids (Method 3580, waste dilution) may be analyzed by direct injection. A 2 to 5 ul aliquot of sample is injected into a gas chromatograph (GC) using the direct injection technique, and compounds in the GC effluent are detected by an electron capture detector (ECD).

2.2 The sensitivity of Method 8082 usually depends on the concentration of interferences rather than on instrumental limitations. If interferences prevent detection of the analytes, Method 8082 may also be performed on samples that have undergone the following cleanups: Method 3660 - Sulfur Cleanup and Method 3665 - Sulfuric Acid Cleanup.

3.0 INTERFERENCES

Interferences by phthalate esters can pose a problem in PCB determinations when using the electron capture detector. Common flexible plastics contain various amounts of phthalates. Care has to be taken to avoid using any plastic materials during the extraction process. Exhaustive cleanup of reagents and glassware may be required to eliminate background phthalate contamination.

Compounds from the sample matrix to which the detector will respond, such as single-component chlorinated pesticides including the DDT series.

4.0 APPARATUS AND MATERIALS

4.1 Gas chromatograph

4.1.1 GC Hewlett Packard 5890 series I or II connected to the Turbochrom or HP Chemstation data system, or equivalent.

4.1.2 Columns - Instruments are configured with a pre-column originating from the injection port, which is connected to a deactivated glass Y splitter that connects two different columns to two detectors. The most commonly used columns are:

RTX-35 30M x 0.53 mm ID, RTX-5 30M x 0.53 MM ID, or RTX-1701 30M x 0.53 mm ID. Equivalent columns can be used.

4.1.3 Detectors: Electron capture detectors (ECD). Note: Two detectors must be employed when using dual columns.

4.2 Volumetric flasks, class A: sizes as appropriate with the ground-glass stoppers.

4.3 Syringes: various sizes for preparing standards and injecting samples on the instrument.

4.4 Vials: various sizes and types including crimp tops.

4.5 Balances: Analytical, 0.0001 g

4.6 Refrigerator for storage of extracts and standards.

5.0 REAGENTS

5.1 Solvents

5.1.1 Hexane: pesticide quality or equivalent for diluting samples and standards.

5.2 Standards

5.2.1 Stock standard solutions: Solutions purchased from suppliers like Restek or other acceptable retailers. Expiration dates are one year from date of opening vial or sooner if manufacturers date is less. Upon receipt, all standards are logged into the appropriate logbook with the date of receipt, expiration date, source, lot number, solvent and concentration of compounds. Standard solutions are stored at 4°C in polytetrafluoroethylene (PTFE)-sealed containers in the dark.

5.2.2 Calibration standards: Prepared through the dilution of the stock standards with hexane. Expiration date is 6 months or sooner. Information is documented in standards prep logbook. The concentrations of the working PCB calibration standards are 0.05 ug/ml, 0.10 ug/ml, 0.25 ug/ml, 1.0 ug/ml, 2.5 ug/ml, and 10.0 ug/ml. The Aroclor 1660 standard also contain the surrogates Tetrachloro-m-xylene (TCX) and Decachlorobiphenyl (DCB) at the respective concentrations: 0.001 ug/ml, 0.002 ug/ml, 0.005 ug/ml, 0.020 ug/ml, 0.050 ug/ml, and 0.20 ug/ml.

5.2.3 Independent Calibration Verification standard (ICV): Prepared through the dilution of the stock standards with hexane. Expiration date is 6 months or sooner. Information is documented in standards prep logbook. The concentration of the ICV PCB standard is 1.0 ug/ml.

5.2.4 DDT Analog Standard: Standard containing the DDT series in hexane at 0.05 ug/ml.

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

Extracts must be stored under refrigeration and analyzed within 40 days of extraction.

Note: The holding time above is a recommendation. PCBs are very stable in a variety of matrices, and holding times under the conditions listed above may be as long as a year.

7.0 PROCEDURES

7.1 Extraction

Refer to the appropriate SOPs for the correct extraction procedure. In general, water samples are extracted using methods 3510, 3520 or 3535 while solid samples use methods 3540, 3545, or 3550. Tissue samples are extracted using method 3545. Wipes and oils are generally extracted using method 3580.

7.2 Instrument conditions

Refer to the instrument logbook for the current column and conditions.

Typical conditions are:

Makeup flow: 60 ml/min Ar/Methane or Nitrogen
Column flow: 6 ml/min
Injector Temp: 200
Detector Temp: 300
Oven Ramp: 160(0) - 5/min - 260(10)
Run time: 30 min
Injection size: 2 ul

7.3 Calibration

7.3.1 The GC system is calibrated using the external standard calibration procedure. Six-point calibration standards of Aroclor 1660 (Aroclor 1016 and Aroclor 1260),

Aroclor 1242, Aroclor 1248 and Aroclor 1254 are prepared along with mid-point calibration standards of Aroclor 1221 and Aroclor 1232. If Aroclor 1221 or Aroclor 1232 are suspected, then a six-point curve of the respective Aroclor will be analyzed prior to the analysis and quantitation of the sample.

Each calibration standard is injected using the technique that is used to introduce the actual samples into the GC. Three to five characteristic peaks from each Aroclor are used to calibrate a curve. The Target system will calculate a peak height for all three to five peaks in each Aroclor. A separate calibration curve for each of the three to five peaks can be prepared in Target using the peak height against the concentration of the standard. A non-linear calibration applying a second order polynomial (quadratic fit) equation is used to prepare the curve. In order to be used for quantitative purposes, the Coefficient of Determination (r^2) must be greater than or equal to 0.990. The quadratic equation is:

$$y = ax^2 + bx + c$$

where: y = Instrument response
b = Slope of the line
x = Concentration of the calibration standard
c = the intercept

7.3.2 A non-linear calibration model may not be allowable for certain states, federal programs, or clients. South Carolina does not allow non-linear calibration work originating in their state. In these cases, a linear calibration model must be used. Each calibration standard is injected using the technique that is used to introduce the actual samples into the GC. Three to five characteristic peaks from each Aroclor are used to calibrate a curve. The Target system will calculate a peak height for all three to five peaks in each Aroclor. A separate calibration curve for each of the three to five peaks can be prepared in Target using the peak height against the concentration of the standard.

7.3.2.1 Linear calibration using the average calibration factor

The calibration factor (CF) is calculated using the following formula:

$$CF = A_s / C_s$$

Where: A_s = Peak area (or height) of the analyte or surrogate.
C_s = Concentration of the analyte or surrogate, in µg/L.

To evaluate the linearity of the initial calibration, calculate the mean CF, the standard deviation (SD), and the RSD.

If the RSD of the calibration factor is less than or equal to 20% over the calibration range, then linearity through the origin may be assumed, and the average calibration or response factor may be used to determine sample concentrations.

7.3.2.2 Linear calibration using a least squares regression

$$y = bx + c$$

where: y = Instrument response
b = Slope of the line
x = Concentration of the calibration standard
c = the intercept

The analyst should not force the line through the origin, but have the intercept calculated from the five data points. In addition, do not include the origin (0,0) as a sixth calibration point. The regression calculation will generate a correlation coefficient (r) that is a measure of the "goodness of fit" of the regression line to the data. A value of 1.00 indicates a perfect fit. In order to be used for quantitative purposes, r must be greater than or equal to 0.995. The ICAL must be successful before any samples or other QC check samples can be analyzed.

7.3.3 The AR1660 calibration curve must be checked initially by analyzing a standard containing the same analytes as the curve but prepared from another source. If the response of the analytes from the independent source varies by more than ± 20%, a new independent source standard must be analyzed or a new calibration curve must be prepared and/or analyzed.

7.3.4 The working calibration curve must be verified prior to sample analysis and every 10 samples thereafter by injecting the mid-point calibration standard. If the response for any analyte varies from the expected response by more than ± 15%, a new calibration curve must be prepared for that analyte. The average result for 5 peak heights of the Aroclors is used for quantitation.

For clients or projects requiring DoD QSM 4.1, the response for any analyte must not vary from the expected response by more than ± 20%, or a new calibration curve must be prepared for that analyte. If the CCV fails the above criteria, reanalyze all samples since the last successful calibration verification. If reanalysis cannot be performed, data must be qualified and explained in the narrative. Additionally, apply a Q-flag to all results for the specific analyte(s) in all samples since the last acceptable calibration verification.

7.4 Retention time windows

7.4.1 Three injections are made of all the PCBs throughout the course of a 72 hour period.

7.4.2 A major peak from the envelope is chosen and a standard deviation is calculated using the three retention times for that peak.

7.4.3 Plus or minus three times the standard deviation of the retention times for each standard is used to define the retention time window; however, the experience of the analyst should weight heavily in the interpretation of chromatograms. The analyst should use the retention time window, but should primarily rely on pattern recognition.

7.4.4 Retention time windows are calculated for each standard on each GC column at method setup and after major maintenance, including whenever a new GC column is installed. The data is kept on file in the laboratory.

7.4.5 If the calculated retention time window results in a value of 0.03 minutes or less, the laboratory will apply nominal windows. This is done in order to avoid any false negative hits because of the window being to narrow. The windows are: ± 0.07 for all target analytes. By utilizing these windows, a false positive hit may be initially indicated, but an experienced analyst could determine a false positive from scrutinizing the chromatograms. Please note that the use of nominal retention time windows may not be allowable for certain states, federal programs, or clients. South Carolina does not allow the use of nominal limits for compliance work originating in their state. In these cases, a window of ± 0.03 minutes must be used if the established retention time window is less than 0.03 minutes.

7.5 DDT Analog standard; This standard should be analyzed to determine if the commonly found DDT analogs (DDT, DDE, DDD) elute at the same retention times of any of the target PCBs. This standard should be analyzed in conjunction with the retention time window determination.

7.6 Gas chromatographic analysis

7.6.1 Shake samples and let them sit for one minute before vialing for analysis.

7.6.2 All instrument injections are performed using the direct injection technique with an autosampler set for 2-5 ul injection volumes.

7.6.3 Samples are analyzed in a set referred to as an analysis sequence. The sequence begins with instrument calibration as listed in section 7.3 followed by

sample extracts interspersed with mid-concentration calibration standards. Before any samples are analyzed the instrument must be calibrated by analyzing a six-point calibration or a 1.0ppm concentration standard (CV-calibration verification standard) for Aroclor 1660, Aroclor 1242, Aroclor 1248 and Aroclor 1254. If a CV is run, the calculated concentration must not exceed a difference of ± 15%. For clients or projects requiring DoD QSM 4.1, the response for any analyte must not vary from the expected response by more than + 20%, or a new calibration curve must be prepared for that analyte. If the CCV fails the above criteria, reanalyze all samples since the last successful calibration verification. If reanalysis cannot be performed, data must be qualified and explained in the narrative. Additionally, apply a Q-flag to all results for the specific analyte(s) in all samples since the last acceptable calibration verification. Each sample analysis must be bracketed with an acceptable initial calibration or an opening CV and an ending CV for each 12-hour shift. The closing CV for Aroclor 1660 is a 0.25ppm concentration standard. All other Aroclors at the closing of the run remain at 1.0ppm concentration. If a second window of samples is run immediately after the closing CVs, the concentration of Aroclor 1660 at the completion of this window would be 1.0ppm. The calibration standard must also be injected at intervals of not less than once every ten samples and at the end of the analysis sequence. If the CV fails, the instrument is checked for any obvious problems and maintenance is performed if deemed necessary. Another CV is analyzed or the instrument is recalibrated and then samples are injected. All samples that were injected after the standard exceeding the criterion must be reinjected to avoid errors in quantitation, if the initial analysis indicated the presence of the specific target analyte that exceeded the criterion.

7.6.3.1 However, if the standard analyzed after a group of samples exhibits a response for an analyte that is above the acceptance limit, i.e. >15%, and the analyte was not detected in the specific samples analyzed during the analytical shift, then the extracts for those samples do not need to be reanalyzed, as the CV standard has demonstrated that the analyte would have been detected were it present. In contrast, if an analyte above the QC limits was detected in a sample extract, then re-injection is necessary to ensure accurate quantitation. If an analyte was not detected in the sample and the standard response is more than 15% below the initial calibration response, then re-injection is necessary to ensure that the detector response has not deteriorated to the point that the analyte would not have been detected even though it was present.

7.6.4 The center of the retention time window for each analyte and surrogate is established by using the absolute retention time for each analyte and surrogate from the daily opening calibration verification or initial calibration.

7.6.5 The identification of PCBs is based on agreement between the retention times of peaks in the sample chromatogram with the retention time windows established through the analysis of standards of the target analytes. An analyte is tentatively identified when a peak from a sample falls within the daily retention time window. Each tentative identification must be confirmed using a second GC column of dissimilar stationary phase or using another technique such as GC/MS. If the retention times of the peaks on both columns fall within the retention time windows on the respective columns, then the target analyte identification has been confirmed.

7.6.5.1 An additional criterion is applied for the identification and quantitation of PCBs. Identification is based on the characteristic fingerprint retention time and shape of the major peaks. Major peaks are defined as those peaks in the Aroclor standard that are at least 25% of the height of the largest Aroclor peak. The sample chromatogram is compared to the individual Aroclor standard chromatograms. Once the Aroclor pattern has been identified, a concentration is then calculated in Target.

7.6.5.2 Three to five Aroclor concentrations are calculated using the peak heights of the three to five characteristic peaks of the Aroclor. These three to five concentrations are then averaged to determine the concentration of that Aroclor.

7.6.6 When samples are analyzed from a source known to contain specific Aroclors, the results from a single-column analysis may be confirmed on the basis of a clearly recognizable Aroclor pattern.

7.6.7 If the response for an analyte exceeds the calibration range of the system, the sample must be diluted and reanalyzed.

7.6.8 If peak detection and identification are prevented due to interferences, the hexane extract may need to undergo a cleanup. The extract may be subjected to a sulfur cleanup (method 3660) and/or a sulfuric acid cleanup (method 3665).

Note: Samples routinely receive a sulfuric acid clean up. However, for samples from a known site with a clean matrix, a sulfuric acid clean up may not be performed. Whenever a sample receives a cleanup, the associated QC must also be subjected to the same cleanup(s) and reanalyzed.

7.6.9 When a GC system is determined to be out of control because either a CV cannot pass or a six point calibration does not meet the correlation coefficient criteria, instrument maintenance is likely necessary. Routine instrument maintenance may involve changing the septum, replacing the liner, clipping the

pre-column, or replacing the column. This information is recorded in the instrument run log (Figure 1). When an instrument requires more severe maintenance like replacing the ECD or an electronic board, this information is written in the instrument maintenance logbook.

7.7 Calculations

7.7.1 The concentration of an analyte is calculated by using the calibrated curve that is prepared in Target. When an analyte is identified, Target displays a concentration after the file is processed through the appropriate calibration method. Aroclor quantitation is accomplished by the method described in section 7.5.4.1.1. However, if a sample contains more than one Aroclor, a peak common to both analytes must not be used to quantitate either compound.

7.7.2 The concentrations from the reports are then incorporated with the extraction data to arrive at a final concentration.

Water: Concentration (ug/L) = (C) (Vt)/ (Vs)

Soil/Sediment: Concentration (mg/kg) = (C) (Vt)/ (Ws) (D)

where, C = concentration calculated by Target in ug/ml
Vt = Volume of total extract including any instrument dilutions
Vs = Volume of sample extracted
Ws = Weight of sample extracted
D = Decimal total solids

7.8 Data Review

7.8.1 Initial Data Review

The initial data review is accomplished by the analyst who ran the samples. This review is of sufficient quality and detail to provide a list of samples that need to be reanalyzed or diluted and reanalyzed. The initial data review is performed on the detailed quantitation reports of the analyzed samples. This data review examines criteria that directly impact whether or not the sample needs to be reanalyzed and/or extracted. These criteria include:

- QC criteria for method blank, LCS, MS/MSD, and calibration – refer to section 8.0.
- Surrogate recovery
- Chromatography: cleanups, manual integration.
- Target compound detection: quantitation, confirmation, false positives.

The requirement of the GC laboratory is that this initial data review be completed no later than the end of the next workday. After the analyst has completed his or her initial data review, the information is then ready to be processed for reporting. Refer to section 7.8.

7.8.2 Surrogate recovery

All recoveries must meet the most recently laboratory established acceptance limits, which are listed on the GC Laboratory Surrogate Acceptance Limit sheet.

The sample is evaluated for recoveries of the two surrogates. If the recovery of one surrogate is within the acceptance limit, and the second is out, the data is narrated. If the surrogate recoveries are high for both and the sample contains less than the PQL for all target analytes, the data is narrated. If the surrogate recoveries are low and may be attributable to matrix interference or a matrix effect, the data is narrated. If the surrogate recoveries are low and the sample concentration is less than the PQL for all target analytes and there is no apparent matrix effect, reextract the sample.

For method blanks, if the recoveries of both surrogates are low or high, and the blank does not contain any target analytes above the PQL, and the recoveries of both surrogates in the sample(s) are acceptable, the data is narrated. If the recoveries in the blank are low and it does not contain any target analytes above the PQL, and the recoveries in the samples are acceptable but the sample contains one or more target analytes above the PQL, the sample may be reextracted.

For laboratory control samples (LCS), if the only discrepancy in the extraction batch is with the LCS, and the analyte spike recoveries are acceptable, the data is narrated. If the recoveries of both the surrogates and the analyte spikes are low, the samples may need to be reextracted.
For DoD QSM 4.1, use QC acceptance criteria specified by DoD, if available. Otherwise, use in-house control limits. When the surrogate recoveries fall outside of the acceptance criteria, apply Q-flag to all associated analytes.

7.8.3 Chromatography

The chromatography should be examined for the presence of any non-target peaks, which can be used as an indication of whether or not matrix interference might be influencing surrogate recoveries. If the chromatogram indicates interferences, then a cleanup may be needed. See section 7.5.7.

Manual integrations are to be performed when chromatographic conditions preclude the computer algorithm from correctly integrating the peak of concern. In no instance shall a manual integration be performed solely to bring a peak within criteria.
Each peak of concern is examined by the primary analyst to ensure that the peak was integrated properly by the computer algorithm. Should a manual integration be necessary (for instance, due to a split peak, peak tailing, or incomplete resolution of isomeric pairs), an "m" qualifier will automatically be printed on the quantitation report summary. The analyst will date and initial the "m" on the quanitation report summary and assign a code that indicates the reason for the manual integration. Refer to Katahdin SOP QA-812 "Manual Integration on GC/MS, GC, HPLC and IC Datasystems" for more information.

7.8.4 Target Compound Detection

GC analysis relies heavily on the experience of the analyst. Sample chromatograms must be evaluated focusing on scientific judgment, knowledge of the column behavior and matrix effects. The chromatogram from channel A is evaluated with that from channel B. If a target analyte is present on both channels and the concentration is within the calibration range, and the quantitation from both chromatograms agrees within ±40%, the analyte is considered present in the sample. In cases where the RPD is greater than 40% and the analyte is reported, the analyte must be J-flagged and narrated. The higher of the two concentrations is reported unless matrix interference is causing erroneously high results. In this case report the lower result and narrate. In some cases a non-confirming analyte may be reported. In these cases the analyte must be Q-flagged and narrated...

In order to avoid reporting false positives, identified peaks on a chromatogram may need to be undetected electronically in Target. The possible scenarios are: If an analyte is present on one column but its concentration is below the PQL, if an analyte is present on one column but does not confirm on the other channel, if an analyte is present on both columns but the concentrations differ by more than 40%, or if an analyte is present but its retention time is ±0.04 minutes or more than the retention time of the analyte in the preceding CV. The GC Analyst must rely on technical experience in reviewing chromatograms in determining if a hit is an actual analyte or a false positive.

If reporting data that has an RPD that is >40%, the data must be flagged with a "J" indicating that the result is an estimated value. Sometimes interference on one column (i.e. sulfur) will prevent a target analyte from detection and it is present on the conformational column. In this scenario, the result would be

reported from one column and need to be "Q" flagged to indicate that it was not confirmed on a second column.

7.8.5 Reporting

After the chromatograms have been reviewed and any target analytes have been quantitated using Target, the necessary files are brought into QuickForms. Depending on the QC level requested by the client, a Report of Analysis (ROA) and additional reports, such as LCS forms and chronology forms, are generated. The package is assembled to include the necessary forms and raw data. The data package is reviewed by the primary analyst and then forwarded to the secondary reviewer. The secondary reviewer validates the data and checks the package for any errors. When completed, the package is sent to the Department Manager for final review. A completed review checklist is provided with each package. The final data package from the Organics department is then processed by the Data Management department.

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

Refer to Table 1 and to details in this section for a summary of QC requirements, acceptance criteria, and corrective actions. These criteria are intended to be guidelines for analysts. The criteria does not cover all possible situations. If any of the QC requirements are outside the recovery ranges listed in this section or in Table 1, all associated samples must be evaluated against all the QC. In some cases data may be reported, but may be reanalyzed in other cases. Making new reagents and standards may be necessary if the standardization is suspect. The corrective actions listed in this section and in Table 1 may rely on analyst experience to make sound scientific judgments. These decisions are based on holding time considerations, client and project specific Data Quality Objectives and on review of chromatograms. The supervisor, Operations Manager, and/or Quality Assurance Officer may be consulted to evaluate data. Some samples may not be able to be reanalyzed within hold time. In these cases "qualified" data with narration may be advisable after consultation with the client.

In some cases the standard QC requirements listed in this section and in Table 1 may not be sufficient to meet the Data Quality Objectives of the specific project. Much of the work performed at the lab is analyzed in accordance with specific QC requirements spelled out in a project specific Quality Assurance Project Plan (QAPP) or in a program specific Quality Systems Manual (QSM). The reporting limits, acceptance criteria and/or corrective actions may be different than those specified in this SOP. In these cases the appropriate information will be communicated to the Department Manager and/or senior chemists before initiation of the analyses so that specific product codes can be produced for the project. In addition, the work order notes for each project will describe the specific QAPP or QSM to be followed.

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8.1 For each analytical batch (up to 20 samples), a method blank, laboratory control sample (LCS), matrix spike and matrix spike duplicate are analyzed. They are carried through all stages of the sample preparation and analysis steps.

8.2 Spike concentrations: The LCS and the MS/MSD are spiked at the same concentration with Aroclor 1660. The spike concentrations are:

Compound	WATER ug/L	SOILS mg/kg
Aroclor 1660	5.0	0.17

The surrogate spike concentrations in the final extract are:

Compound	WATER ug/ml	SOILS ug/ml
Tetrachloro-m-xylene(TCX)	0.10	0.10
DCB	0.10	0.10

8.3 LCS and MS/MSD acceptance criteria and Corrective Action: All QC samples are calculated for percent recovery of the spiked analyte. The recoveries are compared to laboratory established acceptance limits. The LCS acceptance limits for PCBs are established for both water and soil matrices. The MS/MSD acceptance limits for PCBs use the respective matrix LCS acceptance limits. Separate limits for MS/MSD pairs are not calculated because of the varying matrices involved. In addition many of the MS/MSD data points cannot be used (i.e. recoveries not calculable due to a matrix effect).

If any spike compound in the laboratory control sample falls outside of the established recovery acceptance limit window, the QC sample is considered to be out of control and any sample that is associated should be evaluated with other QC elements to determine the corrective action. If the recovery is high and the associated samples do not contain the specific compound(s), the data can possibly be accepted with narration. In other cases, the associated samples must be extracted.
If a spike compound is outside of the acceptance limits in the matrix spike sample but is acceptable in the LCS, the data is considered acceptable. The cause of the failure is possibly attributable to matrix interference. However, if the compound fails in both the LCS and the MS/MSD, the result for that analyte is suspect and may not be reported for regulatory compliance purposes.

For DoD QSM 4.1, use QC acceptance criteria specified by DoD, if available. Otherwise use in-house control limits. In-house control limits must not be greater than $\pm$ 3 times the standard deviation of the mean LCS recovery. If the LCS fails the acceptance criteria, correct problem, then reprep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available. If reanalysis cannot be performed, data must be qualified and explained in

the narrative. Apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch.

For MS, when applying DoD QSM 4.1, apply J-flag to specific analyte(s) also in parent sample, if acceptance criteria not met. RPD must be $\leq$ 30% between MS and MSD.

8.4 Surrogate acceptance criteria and Corrective Action: Surrogate recoveries are calculated on all samples, blanks and spikes. The recoveries are compared to laboratory established acceptance limits.

When a sample has a surrogate that falls outside of the laboratory established acceptance limit window, the problem should be investigated. If the recovery looks like it is affected by the sample matrix, the sample may be reinjected to confirm matrix interference. When a sample has no detectable surrogate recovery, the sample should be reextracted.

For DoD QSM 4.1, use QC acceptance criteria specified by DoD, if available. Otherwise, use in-house control limits. When the surrogate recoveries fall outside of the acceptance criteria, apply Q-flag to all associated analytes.

8.5 CAR: Whenever data is not acceptable because of a failing LCS or surrogate recovery, a corrective action report (CAR) must be initiated as soon as possible to document resolution.

9.0 METHOD PERFORMANCE

The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero. The MDLs are determined prior to sample analysis per type of instrument and filed with the Organic Department Manager and with the QAO. Refer to the current revision of Katahdin SOP QA-806, Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications, for procedures on determining the MDL.

Refer to the current revision of Method 8082 for other method performance parameters and requirements.

10.0 APPLICABLE DOCUMENTS/REFERENCES

Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition. Final Update IV, dated February, 2007, Method 8082A.

TITLE: ANALYSIS OF PCBs AS TOTAL AROCLORS BY GAS CHROMATOGRAPHY/ ELECTRON CAPTURE DETECTOR (GC/ECD): SW-846 METHOD 8082

Department of Defense Quality Systems Manual for Environmental Laboratories (DoD QSM), Version 4.1, 04/22/09.

The National Environmental Laboratory Accreditation Conference (NELAC) Standards, June 2003.

Katahdin Analytical Services, Inc., SOP CA-101, Equipment Maintenance and Troubleshooting, current revision.

Katahdin Analytical Services, Inc., SOP CA-106, Standard Preparation, Documentation and Traceability, current revision.

Katahdin Analytical Services, Inc., SOP CA-500, Preparation of Soil/Sediment Samples by Sonication Using Method 3550 for Subsequent Pesticides/PCBs Analysis, current revision.

Katahdin Analytical Services, Inc., SOP CA-515, Preparation of Aqueous Samples for Pesticides/PCBs Analysis-Methods 3510 and 3520, current revision.

Katahdin Analytical Services, Inc., SOP CA-524, Preparation of Soil/Sediment Samples by Soxhlet Extraction Using Method 3540 for Subsequent Pesticides/PCBs Analysis, current revision.

Katahdin Analytical Services, Inc., SOP QA-806, Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications, current revision.

LIST OF FIGURES

TITLE: ANALYSIS OF PCBs AS TOTAL AROCLORS BY GAS CHROMATOGRAPHY/ ELECTRON CAPTURE DETECTOR (GC/ECD): SW-846 METHOD 8082

TABLE 1

QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
6pt calibration of Aroclor 1660, 1242, 1248, 1254 and mid-point cal of other Aroclors	Initial cal prior to sample analysis	6 pt calibration – Coefficient of Determination (r^2) ≥ 0.990	(1) Repeat Initial calibration (2) If single pt cal Aroclor is identified in analysis of sample, 6-pt calibration run of identified compound with reanalysis of sample.
5pt calibration of Aroclor 1660, 1242, 1248, 1254 and mid-point cal of other Aroclors	Initial cal prior to sample analysis	5 pt calibration – (r) ≥ 0.990	(1) Repeat Initial calibration (2) If single pt cal Aroclor is identified in analysis of sample, 6-pt calibration run of identified compound with reanalysis of sample.
Independent Calibration Verification	Immediately following calibration	± 20 % D	(1) Reanalyze standard (2) Reprep standard (3) Reprep standard from fresh stock.
CCV	After every 10 samples; If calibration curve previously analyzed, analyze daily before samples.	± 20 % D	(1) Evaluate the samples: If the %D >+15% and sample results are <PQL, narrate. (2) If %D >±15% only on one channel, narrate. If %D >±15% for closing CV, and is likely a result of matrix interference, narrate. (3) Otherwise, reanalyze all samples back to last acceptable CV.
Method blank	One per prep batch	No analyte detected >PQL	(1) Investigate source of contamination (2) Evaluate the samples and associated QC: i.e. If the blank results are above the PQL, report sample results which are <PQL or > 10X the blank concentration. (3) Otherwise, reprep a blank and the remaining samples.
LCS	One per prep batch of twenty or fewer samples	± 15% D	(1) Evaluate the samples and associated QC: i.e. If an MS/MSD was performed and acceptable, narrate. (2) If an LCS/LCSD was performed and only one of the set was unacceptable, narrate. (3) If the surrogate recoveries in the LCS are also low but are acceptable in the blank and samples, narrate. (4) If the LCS recovery is high but the sample results are <PQL, narrate. (5) Otherwise, reprep a blank, QC and the remaining samples.
Matrix Spike\ Matrix Spike Duplicate	One for every set of 20 samples	Same as for LCS	(1) Evaluate the samples and associated QC: i.e. If the LCS results are acceptable, narrate. (2) If both the LCS and MS/MSD are unacceptable reprep samples and QC.

TITLE: ANALYSIS OF PCBs AS TOTAL AROCLORS BY GAS CHROMATOGRAPHY/ ELECTRON CAPTURE DETECTOR (GC/ECD): SW-846 METHOD 8082

TABLE 1 (cont.)

QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Sample Duplicate	One sample duplicate per ten samples if requested	RPD $\leq$20	(1) If lab QC in criteria and matrix interference suspected, flag data or narrate (2) Otherwise, reanalyze
Demonstration of analyst proficiency – 4 replicates	Once per analyst initially and annually thereafter	P&A meet method criteria	(1) Repeat P&A study
MDL study	Refer to KAS SOP QA-806, "Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications", current revision.		

TITLE: ANALYSIS OF PCBs AS TOTAL AROCLORS BY GAS CHROMATOGRAPHY/ ELECTRON CAPTURE DETECTOR (GC/ECD): SW-846 METHOD 8082

TABLE 2

DOD QSM VERSION 4.1 QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Demonstrate acceptable analytical capability	Prior to using any test method and at any time there is a significant change in instrument type, personnel, test method, or sample matrix.	QC acceptance criteria published by DoD, if available; otherwise, method-specified criteria.	Recalculate results; locate and fix problem, then rerun demonstration for those analytes that did not meet criteria.	Not Applicable (NA).	This is a demonstration of analytical ability to generate acceptable precision and bias per the procedure in Appendix C. No analysis shall be allowed by analyst until successful demonstration of capability is complete.
LOD determination and verification	Refer to current revision of SOP QA-806				
LOQ establishment and verification	Refer to current revision of SOP QA-806				
Retention time (RT) window width calculated for each analyte and surrogate	At method set-up and after major maintenance (e.g., column change).	RT width is ± 3 times standard deviation for each analyte RT from a 72-hour study.	NA.	NA.	
Minimum five-point initial calibration (ICAL) for all analytes	ICAL prior to sample analysis.	6 point calibration of Aroclors 1660, 1242, 1248, and 1254 - One of the options below: Option 1: RSD for each analyte ≤ 20%; Option 2: linear least squares regression: r ≥ 0.995; Option 3: non-linear regression: coefficient of determination (COD) r2 ≥ 0.99 (6 points shall be used for second order). Mid point calibration of Aroclors 1221 and 1232; if targets are detected, 6-point calibration is performed.	Correct problem then repeat ICAL.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be run until ICAL has passed. Calibration may not be forced through the origin. Quantitation for multicomponent analytes such as chlordane, toxaphene, and Aroclors must be performed using a 5-point calibration. Results may not be quantitated using a single point.
Retention time window position establishment for each analyte and surrogate	Once per ICAL and at the beginning of the analytical shift.	Position shall be set using the midpoint standard of the ICAL curve when ICAL is performed. On days when ICAL is not performed, the initial CCV is used.	NA.	NA.	

TITLE: ANALYSIS OF PCBs AS TOTAL AROCLORS BY GAS CHROMATOGRAPHY/ ELECTRON CAPTURE DETECTOR (GC/ECD): SW-846 METHOD 8082

TABLE 2 (cont)

DOD QSM VERSION 4.1 QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Second source calibration verification (ICV)	Immediately following ICAL.	All project analytes within established retention time windows. GC methods: All project analytes within ± 20% of expected value from the ICAL.	Correct problem, rerun ICV. If that fails, repeat ICAL.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be run until calibration has been verified.
calibration verification (CCV)	Prior to sample analysis, after every 10 field samples, and at the end of the analysis sequence.	All project analytes within established retention time windows. GC methods: All project analytes within ± 20% of expected value from the ICAL.	Correct problem, then rerun calibration verification. If that fails, then repeat ICAL. Reanalyze all samples since the last successful calibration verification.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to all results for the specific analyte(s) in all samples since the last acceptable calibration verification.	Problem must be corrected. Results may not be reported without a valid CCV. Flagging is only appropriate in cases where the samples cannot be reanalyzed. Retention time windows are updated per the method.
Method blank	One per preparatory batch.	No analytes detected > ½ RL (> RL for common lab contaminants) and > 1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater). Blank result must not otherwise affect sample results.	Correct the problem. Report sample results that are <LOD or >10x the blank concentration. Reprepare and reanalyze the method blank and all associated samples with results > LOD and < 10x the contaminated blank result. Contact Client if samples cannot be reprepped within hold time.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply B-flag to all results for the specific analyte(s) in all samples in the associated preparatory batch.	Problem must be corrected. Results may not be reported without a valid method blank. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Laboratory control sample (LCS) containing all analytes to be reported, including surrogates	One per preparatory batch.	The laboratory shall use laboratory control limits (CLs) or use DoD-generated LCS-CLs, if available depending on project requirements. In-house CLs may not be greater than ± 3 times the standard deviation of the mean LCS recovery. A number of analytes may fall outside the CL but within marginal exceedance limit depending on the total number of analytes in the LCS.	Correct problem, then reprep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available. Refer to Table G-1 for number of marginal exceedences allowed. Contact Client if samples cannot be reprepped within hold time.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch.	Problem must be corrected. Results may not be reported without a valid LCS. Flagging is only appropriate in cases where the samples cannot be reanalyzed.

TITLE: ANALYSIS OF PCBs AS TOTAL AROCLORS BY GAS CHROMATOGRAPHY/ ELECTRON CAPTURE DETECTOR (GC/ECD): SW-846 METHOD 8082

TABLE 2 (cont.)

DOD QSM VERSION 4.1 QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Matrix spike (MS)	One per preparatory batch per matrix if sufficient sample is available.	For matrix evaluation, use LCS acceptance criteria.	Examine the project-specific DQOs. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met.	For matrix evaluation only. If MS results are outside the LCS limits, the data shall be evaluated to determine the source of difference and to determine if there is a matrix effect or analytical error.
Matrix Spike duplicate (MSD)	One per preparatory batch per matrix if sufficient sample is available.	MSD: For matrix evaluation, use LCS acceptance criteria. MS/MSD: RPD ≤ 30%.	Examine the project-specific DQOs. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met.	The data shall be evaluated to determine the source of difference.
Surrogate spike	All field and QC samples.	The laboratory shall use laboratory control limits (CLs) or use DoD-generated LCS-CLs, if available depending on project requirements.	For QC and field samples, correct problem then reprep and reanalyze all failed samples for failed surrogates in the associated preparatory batch, if sufficient sample material is available. If obvious chromatographic interference with surrogate is present, reanalysis may not be necessary. Contact Client if samples cannot be reprepped within hold time.	Apply Q-flag to all associated analytes if acceptance criteria are not met.	Alternative surrogates are recommended when there is obvious chromatographic interference.
Confirmation of positive results (second column or second detector)	All positive results must be confirmed (with the exception of Method 8015).	Calibration and QC criteria same as for initial or primary column analysis. Results between primary and second column RPD ≤ 40%.	NA.	Apply J-flag if RPD > 40%. Discuss in the case narrative.	Use project-specific reporting requirements if available; otherwise, use method reporting requirements; otherwise, report the result from the primary column (see Box D-16).
Results reported between DL and LOQ	NA.	NA.	NA.	Apply J-flag to all results between DL and LOQ.	

TITLE: ANALYSIS OF PCBs AS TOTAL AROCLORS BY GAS CHROMATOGRAPHY/ ELECTRON CAPTURE DETECTOR (GC/ECD): SW-846 METHOD 8082

TABLE 3

SUMMARY OF METHOD MODIFICATIONS

TOPIC	KATAHDIN SOP CA-329-11	METHOD 8082, current revision
Procedures	7.4.5 If the calculated retention time window results in a value of 0.03 minutes or less, the laboratory will apply nominal windows. This is done in order to avoid any false negative hits because of the window being to narrow. The windows are: ± 0.07 for all target analytes. By utilizing these windows, a false positive hit may be initially indicated, but an experienced analyst could determine a false positive from scrutinizing the chromatograms. Please note that the use of nominal retention time windows may not be allowable for certain states, federal programs, or clients. South Carolina does not allow the use of nominal limits for compliance work originating in their state. In these cases, a window of ± 0.03 minutes must be used if the established retention time window is less than 0.03 minutes.	9.3 refers to method 8000B section 7.6.3: If the standard deviation of the retention times for a target compound is 0.000 (i.e., no difference between the absolute retention times), then the laboratory may either collect data from additional injections of standards or use a default standard deviation of 0.01 minutes. (Recording retention times to three decimal places rather than only two should minimize the instances in which the standard deviation is calculated as 0.000).
Apparatus/Materials		
Reagents		
Sample Preservation and handling		
QC – Spikes		
QC – LCS		
QC – Accuracy/ Precision		
QC - MDL	PQL Practical Quantitation Level – three to ten times the MDL.	EQL Estimated Quantitation Level – five to ten times the MDL

TITLE: ANALYSIS OF PCBs AS TOTAL AROCLORS BY GAS CHROMATOGRAPHY/ ELECTRON CAPTURE DETECTOR (GC/ECD): SW-846 METHOD 8082

FIGURE 1

EXAMPLE OF INSTRUMENT RUN LOG

Katahdin Analytical Services, Inc.
GC Laboratory Instrument Runlog
Instrument: GC06
Amount Injected 2 uL

Method: SW846 8082 or EPA 608
Reviewed by/ Date:

Date	Init.	Result File	Sample ID	Y/N	Method	Column	Comments
7.31.07	SJL	GAG3/4152	WG41666-3 3510	Y	PCB/8074A	298/299	
		153	Hexane	N			
		154	AR1660 0.25	Y			↑DC6 ↑1260 both P4274
		155	AR1242 1.0	Y			P4274
		156	AR1248				P4275
		157	AR1254				P4276
		158	SA3963-1 3540				
		159	SA3987-1				
		160	-2				
		161	-3				
		162	-4				
		163	SA3978-1				
		164	-2				
		165	-3				
8.1.07		166	-4				
		167	-5	N			needs SC.
		168	Hexane				
		169	AR1660 1.0	Y			P4273
		170	AR1242				P4274
		171	AR1248				P4275
		172	AR1254				P4276
		173	SA3978-6 3540	N			needs SC
		174	SA3958-4 3510	Y			
		175	-6				
		176	WG41667-1				
		177	-2				
		178	-3				
		179	-4				
		180	~~SA3958-5~~				
		181	SA3958-1				
		182	-2				
		183	Hexane	N			

QAGC167 0000085

TITLE: ANALYSIS OF PCBs AS TOTAL AROCLORS BY GAS CHROMATOGRAPHY/ ELECTRON CAPTURE DETECTOR (GC/ECD): SW-846 METHOD 8082

FIGURE 2

DATA REVIEW CHECKLIST

PRIMARY REVIEW CHECKLIST

Verbal Due Date _____ **(** ☐ **Verbals turned in). DueDate**_____

Client:		Primary	Secondary
Method:		Date:	Date:
SDG No:	Level:	Initials:	Initials:
KAS No:			Approved : ☐ Yes

DODQSM 3.0 ☐ **DODQSM 4.1** ☐ **WITH LAB. LIMITS** ☐
QUAPP ☐ **LAB** ☐ **(** *REPORT* ***ND's to MDL*** ☐ **)**

List all curves that are scanned. _____ _____

_____ _____ _____ _____

Narrate which QC limits were used for (Surr., LCS,s MS/MSDs.) _____

All needed forms are present . _____

Correct Work Order Number or SDG name (all forms). _____

Correct project name and spelling (all forms). (Truncated ☐). _____

Correct file numbers (all forms). _____

Analysis Date Correct. _____

Extraction Method & Analysis Method Correct. _____

Product list compared to ROAs (compounds & PQLs). _____

Chromatogram reviewed for unlabeled peaks (check product list). _____

Flagging of all ROAs correct (Florida Flagging ☐). _____

All tunes included (level IV) . _____

All log book pages included (Soil weights,TCLP & SPLP). _____

Verify DOD QSM criteria. _____

Narrate any method deviations. (Blanks, LCS,s etc.) _____

Sign & Date Manual integration **(Narrate as needed).** _____

Sample I.D's Truncated (**NARRATED**). YES ☐ Please list KAS # below :

_____

QA-044 – Revision 1 – 04/05/2010

TITLE: ANALYSIS OF PCBs AS TOTAL AROCLORS BY GAS CHROMATOGRAPHY/ ELECTRON CAPTURE DETECTOR (GC/ECD): SW-846 METHOD 8082

FIGURE 3

PQLs FOR METHOD 8082

ANALYTE	Practical Quantitation Level (PQL) (ug/L)	Practical Quantitation Level (PQL) (ug/kg)
PCB-1016	0.50	17
PCB-1221	0.50	17
PCB-1232	0.50	17
PCB-1242	0.50	17
PCB-1248	0.50	17
PCB-1254	0.50	17
PCB-1260	0.50	17

TITLE: DETERMINATION OF PETROLEUM RANGE ORGANICS (PRO) BY FLORIDA DEPARTMENT OF ENVIRONMENTAL PROTECTION METHOD # FL-PRO

Prepared By: Peter Lemay Date: 7/18/01

Approved By:

Group Supervisor: Peter Lemay Date: 7/18/01

Operations Manager: Dennis Neiman by John Burdick Date: 7/18/01

QA Officer: Deborah J. Nadeau Date: 7/18/01

General Manager: Dennis F. Neiman Date: 7/18/01

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
01	Added definitions and information for new data processing system. Added or changed wording to clarify sections 6 and 7 and Table 2. Added wording to sections 8 and 9 per recent NELAC & Navy audits. Minor changes throughout. New figures 1 and 2.	HRC	11.15.04	11.15.04
02	Changed LIMS to KIMS Sodium Sulfate is purified at vendor added wording to sect. 7.7.2 to clarify	LAD	03/06	03/06
03	Many changes made throughout, including but not limited to, waste management, CV frequency, Spike amounts, statistically derived QC limits and method modifications. Please refer to the QAM/SOP change form filed with the SOP in QA for a detailed list of changes	LAD	09/07	09/07
04	Removed Apparatus and Reagents that are not used. Updated surrogate information	LAD	09/08	09/08

TITLE: **DETERMINATION OF PETROLEUM RANGE ORGANICS (PRO) BY FLORIDA DEPARTMENT OF ENVIRONMENTAL PROTECTION METHOD # FL-PRO**

Please acknowledge receipt of this standard operating procedure by signing and dating both of the spaces provided. Return the bottom half of this sheet to the QA Department.

I acknowledge receipt of copy ____ of document **SOP CA-333-04**, titled **DETERMINATION OF PETROLEUM RANGE ORGANICS (PRO) BY DEPARTMENT OF ENVIRONMENTAL PROTECTION METHOD # FL-PRO**

Recipient: _____ Date: _____

KATAHDIN ANALYTICAL SERVICES, INC.
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy ____ of document **SOP CA-333-04**, titled **DETERMINATION OF PETROLEUM RANGE ORGANICS (PRO) BY FLORIDA DEPARTMENT OF ENVIRONMENTAL PROTECTION METHOD # FL-PRO**

Recipient: _____ Date: _____

TITLE: **DETERMINATION OF PETROLEUM RANGE ORGANICS (PRO) BY FLORIDA DEPARTMENT OF ENVIRONMENTAL PROTECTION METHOD # FL-PRO**

1.0 SCOPE AND APPLICATION

The purpose of this SOP is to describe the method used by Katahdin Analytical Services technical personnel to measure the concentration of petroleum range organics (PRO) in water and soil. These compounds correspond to a hydrocarbon range of C_8-C_{40}.

This method is based on a solvent extraction, Gas Chromatography (GC) procedure. The method is designed to measure the petroleum concentration in environmental samples in the above stated C-Range (nominally diesel through motor oils). It cannot be used as an indication of gasoline contamination. Additional analyses may be performed including, but not limited to, analysis of additional reference materials. These additional efforts are not contained within this method.

1.1 Definitions

PETROLEUM HYDROCARBONS: All chromatographic peaks, both resolved and unresolved, eluting between the peak start of n-octane (n-C_8) and the peak end after n-tetracontane (n-C_{40}). Quantitation is based on direct comparison of the area within this range to the total area of the Petroleum Hydrocarbon standard as determined from FID response using baseline-baseline integration.

PETROLEUM HYDROCARBON STANDARD: A 17-component mix of all even numbered normal alkanes from C8 to C40. This standard serves as a quantitation standard and a retention time window defining Petroleum Hydrocarbons.

SAMPLE MATRIX SPIKE: A selected sample from the analytical batch spiked with the Petroleum Hydrocarbon Standard and surrogate standards. The calculated spike recovery shall be used as a control.

LABORATORY CONTROL SAMPLE: Laboratory reagent grade water or standard soil spiked with the Petroleum Hydrocarbon standard and surrogate standards. The calculated spike recovery may be used as a laboratory control.

METHOD DETECTION LIMIT (MDL): Minimum concentration that an analyte can be measured and reported with a 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix containing the analyte. The MDL is determined using EPA Appendix B to Part 136, CFR 40 Ch. 1(7-1-94) using the Student t Test.

KATAHDIN INFORMATION MANAGEMENT SYSTEM (KIMS): A complete multi-user system with the capabilities of integrating laboratory instrumentation, generating laboratory worksheets, providing complete Lab Order status and generating reports. LIMS utilizes these features through a database.

PE NELSON TURBOCHROM: A data acquisition system that is used to collect chromatographic data. The system can also be used to archive raw data files.

TARGET: A software system that combines full processing, reporting and comprehensive review capabilities, regardless of chromatographic vendor and data type.

TARGET DB: An oracle database used to store and organize all Target data files.

QUICKFORMS: A laboratory reporting software for Target and Target DB. The QuickForms report module for Target is preconfigured with generalized forms and US EPA CLP report forms and disk deliverables, which can be customized.

1.2 Responsibilities

This method is restricted to use by, or under the supervision of analysts experienced in the analysis of PRO by Method FL-PRO. Analysts should be skilled in the interpretation of gas chromatograms and their use as a quantitative tool. Each analyst must demonstrate and document their ability to generate acceptable results with this method. Refer to Katahdin SOP QA-805, current revision, "Personnel Training & Documentation of Capability".

It is the responsibility of all Katahdin technical personnel involved in analysis of PRO by Method FL-PRO to read and understand this SOP, to adhere to the procedures outlined, and to properly document their data in the appropriate lab notebook. Any deviations from the test or irregularities with the samples should also be recorded in the lab notebook and reported to the Department Manager or designated qualified data reviewer responsible for this data.

It is the responsibility of the Department Manager to oversee that members of their group follow this SOP, to ensure that their work is properly documented and to initiate periodic review of the associated logbooks.

1.3 Safety

Users of this procedure must be cognizant of inherent laboratory hazards, proper disposal procedures for contaminated materials and appropriate segregation of hazardous wastes. The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical should be treated as a potential health hazard. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Everyone involved with the procedure must be familiar with the MSDSs (material safety data sheets) for all the materials used in this procedure.

Each qualified analyst or technician must be familiar with Katahdin Analytical safety procedures and the Katahdin Hazardous Waste Management Plan and must follow appropriate procedures. These include the use of appropriate personal protective equipment (PPE) such as safety glasses, gloves and lab coats when working with chemicals or near an instrument and not taking food or drink into the laboratory. Each analyst should know the location of a respirator and all safety equipment. Each analyst shall receive a safety orientation from their Department Manager, or designee, appropriate for the job functions they will perform.

1.4 Pollution Prevention/Waste Disposal

Whenever possible, laboratory personnel should use pollution prevention techniques to address their waste generation. Refer to the current revision of the Katahdin Hazardous Waste Management Plan for further details on pollution prevention techniques.

Wastes generated during the preparation of samples must be disposed of in accordance with the Katahdin Hazardous Waste Management Plan and Safety Manual and SOP SD-903, "Sample Disposal," current revision. Expired standards are lab packed, placed in the Katahdin hazardous waste storage area, and disposed of in accordance with this SOP.

Any methylene chloride solvent waste generated during the preparation of standards etc. should be disposed of in the "D" waste stream satellite accumulation area nearest the point of generation. Acetone and hexane are considered flammable waste, and should be disposed of in the "O" waste stream satellite accumulation area nearest the point of generation. FLPRO sample vials are considered "P" waste and should be disposed of in the corresponding satellite waste accumulation area nearest the point of generation. Please refer to the current revision of SOP CA-107 for the location of satellite waste accumulation areas.

2.0 SUMMARY OF METHOD

2.1 One liter of water or a specified quantity of soil (extraction method dependent) is spiked with two surrogates and extracted with Methylene chloride. The water is removed from the extract, concentrated to a volume of 2.0 mL, and treated with silica gel to remove potential organic interferences. An aliquot is injected onto a capillary column gas chromatograph (GC) equipped with a flame ionization detector (FID). Quanitation is based on the detector response compared to a series of normal alkane standards. This method is suitable for the analysis of waters, soils or wastes.

2.2 This method is based in part on USEPA Methods 8000 and 8100, SW-846, "Test Methods for Evaluating Solid Waste", 3rd Edition, Method OA-2, work by the EPA UST Work Group "Measurement of Petroleum Hydrocarbons: Report on activities to Develop a Manual", 1990, Method AK103.0, Revision 2, PUBL-SW-141, July 1993 and the Florida Department of Environmental Protection Technical Advisory Committee for 62-770, F. A. C, Petroleum Contamination Site Cleanup Criteria.

3.0 INTERFERENCES

3.1 Other organic compounds including chlorinated hydrocarbons, phenols, and phthalate esters are measurable. As defined in the method, the PRO results include these compounds. Spills of known specific constituents should be analyzed and quantified by a method specific for those compounds.

3.2 Method interferences are reduced by washing all glassware with hot soapy water and then rinsing it sequentially with tap water, methanol or acetone, and Methylene chloride. Method blanks must be analyzed with each batch to demonstrate that the samples are free from method interferences.

3.3 High purity reagents (pesticide grade or better) must be used to minimize interferences.

3.4 Contamination by carryover can occur whenever high-level and low-level samples are sequentially analyzed. Whenever an unusually concentrated sample is encountered, it should be followed by analysis of a solvent blank to check for cross-contamination.

3.5 Animal and vegetable oil and grease and biogenic terpenes are also measurable if the sample is not cleaned up before analysis. In order to eliminate false positives from these sources, the silica cleanup is a mandatory part of the procedure.

4.0 APPARATUS AND MATERIALS

4.1 Gas Chromatograph: Analytical system complete with gas chromatograph and all required accessories, including a detector, column supplies, recorder, gases, and syringes. A capillary split/splitless injector operating in the splitless mode is recommended. A data system capable of determining peak areas by integrating from baseline to baseline is required.

4.2 Column 1: 30 m x 0.53 mm ID ZB-5, 1.5 micron film thickness (or equivalent). Column 2: 30 m x 0.53 mm ID ZB-1, 1.5 micron film thickness (or equivalent). The

column must be capable of resolving typical diesel components, and the solvent front from C_8. Other columns may be used if all column performance criteria are met.

4.3 Detector: Flame ionization detector (FID).

4.4 Microsyringes: 1 ul, 5 ul, 10 ul, 25 ul, and 100 ul.

4.5 Disposable pipettes: Pasteur.

4.6 2 ml (and larger) vials with Teflon lined caps for storage of extracts.

5.0 REAGENTS

5.1 Solvents: Methylene chloride: Pesticide grade or equivalent. Store away from other solvents.

5.2 Stock Standards: Aliphatic Hydrocarbon standard mix from a vendor like UltraScientific at a concentration of 500 ug/mL in hexane (each of the 17 components from C_8 to C_{40}). A surrogate solution containing n-Triacontane-d_{62} at a concentration of 5000 ug/mL and another surrogate solution containing o-Terphenyl at a concentration of 2000 ug/mL from a vendor like Restek.

5.3 Calibration Standards: The standards are prepared at the following five different concentrations: 200 ug/ml, 100 ug/ml, 50 ug/ml, 20 ug/ml, and 5 ug/ml (per each component). This is equivalent to 85, 340, 850, 1700, and 3400 ug/ml total alkanes in the standards. The concentration of OTP and triacontane-d_{62} must remain at a constant 50 and 300 ug/ml level in all concentration levels.

5.3.1 Transfer the stock standard solution into a Teflon-sealed screw-cap/crimp cap bottle. Store, with minimal headspace, at 6°C or less and protect from light.

5.3.2 Working standards must be replaced after 6 months, or sooner if comparison with check standards indicates a problem.

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

6.1 Whenever possible, samples should be grab samples which are collected directly into the sample container. Sample collection equipment such as bailer or intermediate containers should be avoided (exceptions: collection from monitoring

wells or grab samples in surface water at depth). Unless required by permit, automatic samplers may not be used. Pumps such as bladder pumps or peristaltic pumps shall not be used.

6.2 All sampling equipment which contacts the sample shall be constructed of teflon®, stainless steel or glass. Under no circumstances can flexible PVC tubing, such as tygon®, be used in the purging or sample collection process.

6.3 Water samples shall be collected in a one liter glass container; soils in a glass jar. All containers shall be sealed with a screw cap with teflon® liner. Water samples shall be acidified to a pH of less than 2 with hydrochloric or sulfuric acid (reagent grade or better).

6.4 The samples shall be stored at 4°C (±2°C) from the time of collection until extraction. Extraction shall be performed on waters within seven days of sample collection and on soils within 14 days of sample collection. All analyses must take place within 40 days of extraction.

7.0 PROCEDURES

7.1 Waters are extracted using a separatory funnel or continuous liquid liquid extraction technique. Soils are extracted using a sonication technique. Alternatively, soils may be extracted by a Soxhlet extraction technique. Refer to Katahdin SOP CA-520, current revision, for sample preparation procedures. After the extracts are concentrated, an appropriate volume (usually 1ul) is injected directly into the GC. (Recommend using splitless injection techniques).

NOTE: NaCl may be added to water samples to improve extraction efficiency.

If the sample concentration exceeds the calibration range for PRO an appropriate dilution should be used. An appropriate dilution is one that keeps the response of major constituents (previously saturated peaks) in the linear range of the detector. If an initial dilution does not accomplish this then an intermediate dilution should be performed.

7.2 Gas Chromatography:

7.2.1 Conditions (For both column 1 and 2): Set column temperature to 60°C for 2 minutes, then 10°C/min. to 300°C and hold for 24 min. Set FID Detector to 310°C and injector to 300°C. Conditions may be altered to improve resolution or recovery of petroleum range organics.

7.2.2 Performance Criteria: GC run conditions and columns must be chosen to meet the following criteria:

7.2.2.1 Resolution of C_8 from the solvent front.

7.2.2.2 The column must be capable of separating typical petroleum hydrocarbon components from the surrogates.

7.3 Retention Time Window

7.3.1 Before establishing windows, be certain that the GC system is within optimum operating conditions. Make three injections of the method standard throughout the course of a 72 hour period. Serial injections over less than a 72 hour period result in retention time windows that are too tight.

7.3.2 Calculate the standard deviation of the absolute retention times for the two surrogates, C_8, and C_{40}.

7.3.2.1 The retention time window for individual peaks is defined as a plus or minus three times the standard deviation of the absolute retention time for each component.

7.3.2.2 In those cases where the standard deviation for a particular analyte is zero, the laboratory should use ±0.05 min as a retention time window.

7.3.3 The laboratory must calculate retention time windows for these standards on each GC column and whenever a new GC column is installed. The data are retained by the laboratory.

7.4 PRO Calibration

7.4.1 Initial Calibration – Calibration shall be by external calibration using a minimum of 5 concentration levels for the initial calibration. Quantitation shall be by linear regression.

In all cases, response of the standards must be determined by continuous integration of all responses (excluding surrogates) from a forced baseline beginning at a point prior to the elution of C_8 to a point past C_{40}. All responses must be determined as responses to baseline and not valley to valley. A method is calibrated for all five levels using the area of each of the 17 individual alkanes and the area of the two surrogates and a total area of the Petroleum Hydrocarbon Standard (PRO) (which is the total area of the seventeen alkanes for each level).

7.4.1.1 Linear Regression – The linear regression shall be calculated using the total PRO area versus the PRO concentration. The correlation coefficient shall be equal to or greater than 0.995.

7.4.1.2 The accuracy of the initial calibration shall be verified by injecting a midpoint concentration of a standard mix that has been obtained from a different source. The calculated value shall be within ± 20% of the expected value.

7.4.2 Continuing Calibration – The calibration curve must be verified on each working day by the injection of a continuing calibration standard (CV) at a midpoint concentration. This standard must be evaluated prior to the analysis of samples.

In addition, a continuing calibration must be run every 10 samples and at the end of the sequence. The concentration of these should vary, with at least one at a level of 1-2 times the calculated PQL as a verification of sensitivity. To accomplish this, continuing calibrations at 50 ug/ml and 20 ug/ml (each component) should be ran.

7.4.2.1 If the concentration of this standard varies from the predicted concentration by more than ± 25%, a new initial calibration curve must be prepared and verified before samples are analyzed.

7.4.2.2 Retention Time Window – Establish daily retention time windows for each analyte of interest using the absolute retention time for each analyte as the midpoint of the window for that day **if** after analyzing the midpoint it is determined that one or more analytes falls outside of the previously established absolute retention time window. The daily retention time window equals the midpoint ± three times the standard deviation determined in Section 7.3.

7.5 Gas Chromatography Analysis

7.5.1 A 1 ul injection volume is analyzed by GC/FID.

7.5.2 If an initial calibration has already been performed, verify the calibration by analysis and evaluation of a mid-point CV on each working day.

In addition, a CV must be run every 10 samples and at the end of the sequence.

7.5.3 Evaluate the CV per 7.4.2.1 and 7.4.2.2. If either performance criteria fails, the instrument must be recalibrated and all samples which were injected after the failed standard must be reanalyzed.

7.5.4 A Methylene chloride blank will be run in every sequence to determine the area generated on normal baseline bleed under the conditions prevailing in the 24 hour period if requested by the client. This area is determined by continuous integration of all responses under the same conditions (i.e. forced baseline and predetermined time interval) as the samples. This blank is calculated as the solvent blank and the value should be less than the PQL.

Methylene chloride blanks should also be run after samples suspected of being highly concentrated to prevent carryover. If the blank analysis shows contamination, additional blanks should be analyzed until the system is shown to be free from contaminants.

7.5.5 If the sample concentration exceeds the linear range of the method in the final extract, the extract must be diluted and reanalyzed.

7.5.6 Baseline correction is allowed to correct for rises due to temperature programming. Range integration is corrected by the automatic subtraction of the baseline established by activation of a programmed run without the injection of any material. Instrument baseline must be established for every batch of samples.

7.6 Calculations

7.6.1 The integrated area for all peaks eluting from n-octane through n-tetracontane shall be determined using a baseline drawn from the baseline point to n-octane to a point past n-tetracontane where the baseline returns to normal. All area including the "hump-a-gram" and surrogate standards shall be included. Do not integrate valley to valley for individual peaks except for the two surrogates. The concentration of the PRO is calculated by using the calibrated curve that is prepared in Target. Target displays a concentration when the file is processed through the appropriate calibrated method.

7.6.2 The concentrations from the reports are then incorporated with the extraction data to arrive at a final concentration.

7.6.2.1 Water: Conc (ug/L) = (Amt) (DF) ((Vt/Vo) 1000)

7.6.2.2 Soil/Sediment: Conc (mg/kg) = (Amt) (DF) ((Vt/Vo) (100/(100-M)))

where,

Amt = adjusted concentration calculated by Target in ug/ml
Vt = Volume of total extract
Vo = Volume or weight of sample extracted
M = % Moisture
DF = Dilution Factor

7.7 Data Review

7.7.1 Initial Data Review

The initial data review is accomplished by the analyst who ran the samples. This review is of sufficient quality and detail to provide a list of samples that need to be reanalyzed or diluted and reanalyzed. The initial data review is performed in Target Review. This data review examines criteria that directly impact whether or not the sample needs to be reanalyzed and/or extracted. These criteria include:

- QC criteria for method blank, LCS, MS/MSD, and calibration – refer to section 8.0.
- Surrogate recovery
- Chromatography: manual integration.
- Target compound detection: quantitation, false positives.

The requirement of the GC laboratory is that this initial data review be completed no later than the end of the next work day. After the analyst has completed his or her initial data review, the information is then ready to be processed for reporting. Refer to section 7.8.

7.7.2 Surrogate recovery

The recoveries for o-Terphenyl are compared to the method acceptance limits. The recoveries for n-Triacontane-d_{62} are compared to nominal acceptance limits of 70-130% until laboratory acceptance limits can be established.

The sample is evaluated for recoveries of the surrogate OTP and n-Triacontane-d_{62}. If the recovery is low and there is no apparent matrix effect, the sample should be reanalyzed. If the reanalysis is still low, re-extract. If the recovery is low and there may be a matrix effect, reanalyze to confirm a matrix effect and narrate. If the surrogate is high and the sample results are less than the PQL, or there is likely a matrix effect, narrate.

7.7.3 Chromatography

The chromatography should be examined for the presence of any non-target peaks, which can be used as an indication of whether or not matrix interference might be influencing surrogate recoveries.

Manual integrations are to be performed when chromatographic conditions preclude the computer algorithm from correctly integrating the peak of concern. In no instance shall a manual integration be performed solely to bring a peak within criteria.

In Target Review, each peak of concern is examined by the primary analyst to ensure that the peak was integrated properly by the computer algorithm. Should a manual integration be necessary (for instance, if the sample contains a concentration of PRO which was integrated "valley to valley" instead of a "baseline to baseline"), manual integration is performed in Target Review. A "m" qualifier will automatically be printed on the quantitation report summary indicating that a manual integration was performed. For specific procedures on how to manually integrate, refer to Katahdin SOP QA-811, Manual Integration, current revision.

7.8 Reporting

After the chromatograms have been reviewed and any target analytes have been quantitated using Target, the necessary files are brought into QuickForms. Depending on the QC level requested by the client, a Report of Analysis (ROA) and additional reports, such as LCS forms and chronology forms, are generated. The package is assembled to include the necessary forms and raw data. The data package is reviewed by the primary analyst and then forwarded to the secondary reviewer. The secondary reviewer validates the data and checks the package for any errors. When completed, the package is sent to the Department Manager for final review. A completed review checklist (Figure 2) is provided with each package. The final data package from the Organics department is then processed by the Data Management department.

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

See below or refer to Table 1 for a summary of QC requirements, acceptance criteria, and corrective actions. These criteria are intended to be guidelines for analysts. The table does not cover all possible situations. If any of the QC requirements are outside the recovery ranges listed in Table 1, all associated samples must be evaluated against all the QC. In some cases data may be reported, but may be reanalyzed in other cases. Making new reagents and standards may be necessary if the standardization is suspect. The corrective

actions listed in Table 1 may rely on analyst experience to make sound scientific judgments. These decisions are based on holding time considerations, client and project specific Data Quality Objectives and on review of chromatograms. The Department Manager, Operations Manager, and/or Quality Assurance Officer may be consulted to evaluate data. It may not be possible to reanalyze samples within hold time. In these cases "qualified" data with narration may be advisable after consultation with the client.

In some cases the standard QC requirements listed in this section and in Table 1 may not be sufficient to meet the Data Quality Objectives of the specific project. Much of the work performed at the lab is analyzed in accordance with specific QC requirements spelled out in a project specific Quality Assurance Project Plan (QAPP) or in a program specific Quality Systems Manual (QSM). The reporting limits, acceptance criteria and/or corrective actions may be different than those specified in this SOP. In these cases the appropriate information will be communicated to the Department Manager and/or senior chemists before initiation of the analyses so that specific product codes can be produced for the project. In addition, the work order notes for each project will describe the specific QAPP or QSM to be followed.

8.1 For each analytical batch (up to 20 samples), a method blank, laboratory control sample (LCS), matrix spike and matrix spike duplicate or sample duplicate are analyzed. They are carried through all stages of the sample preparation and analysis steps.

8.2 The laboratory shall generate control limits based on +/-3 standard deviations from the average recovery for all spikes and surrogates, and + 3 standard deviations from the average precision value for all duplicates. The limits that area generated must be within the criteria specified in Table 3 below.

8.3 Spike concentrations: The LCS and the MS/MSD are spiked with the seventeen component PRO mix at the same concentration. The spike concentrations are:

	WATER	SOILS
	ug/L	mg/Kg
PRO	850	28.5

The surrogate spike concentrations in the final extract are:

	WATER	SOILS
	ug/ml	mg/kg
o-Terphenyl	50	1.65
n-Triacontane-d_{62}	300	10

8.4 LCS and MS/MSD acceptance criteria and Corrective Action: All QC samples are calculated for percent recovery of the spiked analyte(s). The recoveries are compared to laboratory established acceptance limits.

If any spike compound in the laboratory control sample falls outside of the established recovery acceptance limit window, the QC sample is considered to be out of control and any sample that is associated should be reextracted. However, if the recovery is high and the associated samples do not contain the specific compound(s), the data can possibly be accepted with narration.

If a spike compound is outside of the acceptance limits in the matrix spike sample but is acceptable in the LCS, the data is considered acceptable. The cause of the failure is possibly attributable to matrix interference. However, if the compound fails in both the LCS and the MS/MSD, the result for that analyte is suspect and may not be reported for regulatory compliance purposes.

8.5 Surrogate acceptance criteria and Corrective Action: Surrogate recoveries are calculated on all samples, blanks and spikes. The recoveries for o-Terphenyl are compared to the method acceptance limits. The recoveries for n-Triacontane-d_{62} are compared to nominal acceptance limits of 70-130% until laboratory acceptance limits can be established.

When a sample has a surrogate that falls outside of the method acceptance limit window, the problem should be investigated. If the recovery looks like it is affected by the sample matrix, the sample may be reinjected to confirm matrix interference. When a sample has no detectable surrogate recovery, the sample should be reextracted.

8.6 CAR: Whenever data is not acceptable because of a failing LCS or surrogate recovery, a corrective action report (CAR) must be initiated as soon as possible.

9.0 METHOD PERFORMANCE

9.1 The MDL of this method is estimated to be at least 4 mg/kg for soil and 0.1 mg/L for water. Each laboratory shall establish a laboratory specific MDL for all matrices prior to analyzing any samples.

9.2 The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero. The MDLs are determined annually per type of instrument and filed with the Organic Department Manager and with the QAO. Refer to the current revision of Katahdin SOP QA-806, Method Detection Limit and Instrument Detection Limit Studies, for procedures on determining the MDL.

Refer to the current revision of the Florida Department of Enviromnental Protection Method for Determination of Petroleum Range Organics (Method # FL-PRO) for other method performance parameters and requirements.

TITLE: **DETERMINATION OF PETROLEUM RANGE ORGANICS (PRO) BY FLORIDA DEPARTMENT OF ENVIRONMENTAL PROTECTION METHOD # FL-PRO**

10.0 APPLICABLE DOCUMENTS/REFERENCES

Florida Department of Environmental Protection, Method for Determination of Petroleum Range Organics, Method # FL-PRO, Revision 1, November, 1995.

ASTM "Standards Methods for Comparison of Waterborne Petroleum Oils by Gas Chromatography," 3328-78.

Wisconsin DNR Modified DRO method, July 1993, Revision 6.

"Test Methods for the Evaluation of Solid Waste: Physical/Chemical Methods", SW-846, third Edition, Final Update III, December 1996, Methods 8000B, 8100, 3500B, 3510C, 3520C, 3540 and 3550B.

LIST OF TABLES AND FIGURES

TITLE: **DETERMINATION OF PETROLEUM RANGE ORGANICS (PRO) BY FLORIDA DEPARTMENT OF ENVIRONMENTAL PROTECTION METHOD # FL-PRO**

TABLE 1

QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Method blank	One per prep batch	No analyte detected >PQL	(1) Investigate source of contamination (2) Evaluate the samples and associated QC: i.e. If the blank results are above the PQL, report sample results which are < PQL or > 10X the blank concentration. Otherwise, reprep a blank and the remaining samples.
LCS	One per prep batch	Laboratory established acceptance limits	(1) Evaluate the samples and associated QC: i.e. If an MS/MSD was performed and acceptable, narrate. If an LCS/LCSD was performed and only one of the set was unacceptable, narrate. If the surrogate recoveries in the LCS are low but are acceptable in the blank and samples, narrate. If the LCS recovery is high but the sample results are < PQL, narrate. Otherwise, reprep a blank and the remaining samples.
Initial Calibration	Initial cal prior to sample analysis	Correlation coefficient => 0.995	(1) Perform instrument maintenance as needed. (2) Reanalyze and or reprep calibration standards.
CV(At or near the midpoint of the ICAL)	On each working day prior to sample analysis if an ICAL was previously analyzed	± 25 %D	(1) Evaluate the samples: If the %D>+25% and sample results are <PQL, narrate. If %D>±25% and is likely a result of matrix interference, narrate. All samples must be reanalyzed that fall within the standard that exceeded criteria and the last standard that was acceptable.
End of sequence CV	At the end of each 12-hour work shift or after running 10 samples, whichever is sooner	± 25 %D	(1) Evaluate sample data if criteria exceeded due to matrix; narrate, and perform maintenance for new samples. (2) If criteria are exceeded and this is not due to matrix, Reanalyze.
Matrix Spike/Matrix Spike Duplicate	One for every set of 20 samples provided samples aliquots are not depleted	Laboratory established acceptance limits RPD< 20 % for waters and < 25 % for solids	(1) Evaluate the samples and associated QC: i.e. If the LCS results are acceptable, narrate. (2) If both the LCS and MS/MSD are unacceptable, reprep the samples and QC.
Sample Duplicate (If required in lieu of MSD)	One sample duplicate per twenty samples	RPD ≤20 for waters, RPD ≤25 for solids	(1) Evaluate data for matrix interference homogeneity of sample.
Demonstration of analyst proficiency; accuracy and precision	One time per analyst initially and annually thereafter	Must pass all applicable QC for method	Repeat analysis until able to perform passing QC; document successful performance in personal training file

TITLE: **DETERMINATION OF PETROLEUM RANGE ORGANICS (PRO) BY FLORIDA DEPARTMENT OF ENVIRONMENTAL PROTECTION METHOD # FL-PRO**

TABLE 1 (cont.)

QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Demonstration of analyst proficiency; accuracy and precision	One time per analyst initially and annually thereafter	Must pass all applicable QC for method	Repeat analysis until able to perform passing QC; document successful performance in personal training file
MDL study	Once per year	Ideally, PQL = at least 3 x MDL	Repeat MDL

TITLE: **DETERMINATION OF PETROLEUM RANGE ORGANICS (PRO) BY FLORIDA DEPARTMENT OF ENVIRONMENTAL PROTECTION METHOD # FL-PRO**

TABLE 2

SUMMARY OF METHOD MODIFICATIONS

TOPIC	KATAHDIN SOP CA-333-04	FL-PRO, current revision
Apparatus/Materials		
Reagents	5.3 Sodium sulfate purified by vendor	7.3 Sodium sulfate purified by heating at 400 deg C for 4 hours or extracting x3 with methylene chloride and drying at 105 deg C
Reagents	5.5 PRO free sand (Muffled)	3.4 Ottawa sand
Sample preservation/ handling		
QC – Method Blank	Table 1 No analyte detected >PQL	10.4 ...TRPH value of the blank shall be at or below the established method detection limit.
QC - Surrogates	Use n-Triacontane-d_{62}. Use nominal limits of 70-130 until laboratory limits can be established.	7.4.1 Recommend C_{39}. Use method acceptance limits.
QC - Spikes	8.2 ...PRO concentration of 850 ug/L for waters and 28.5 mg/kg for soils.	7.4.4 Total PHS concentration in the spiked sample of 5 mg/L in water or 300 mg/kg in soils. ...The concentration of the spike in the sample should be approximately 3-5 times the level expected in the sample...level of the spike should be adjusted...
QC - Accuracy/Precision		
QC – LCS		
QC - MDL		
Procedure	7.4.2.2 Retention Time Window – Establish daily retention time windows for each analyte of interest using the absolute retention time for each analyte as the midpoint of the window for that day **if** after analyzing the midpoint it is determined that one or more analytes falls outside of the previously established absolute retention time window. The daily retention time window equals the midpoint ± three times the standard deviation determined in Section 7.3.	9.3.2.2 Retention Time Window – The retention time window for the surrogates and C8 and C40 shall be within the established range ... If they are out of acceptance range, a new initial calibration must be prepared and verified before samples are analyzed.

TITLE: **DETERMINATION OF PETROLEUM RANGE ORGANICS (PRO) BY FLORIDA DEPARTMENT OF ENVIRONMENTAL PROTECTION METHOD # FL-PRO**

TABLE 2, cont'd

SUMMARY OF METHOD MODIFICATIONS

TOPIC	KATAHDIN SOP CA-333-04	FL-PRO, current revision
Procedure	7.5.4 The Methylene chloride blank is analyzed if requested by the client and will be less than the PQL.	9.5.4 The methylene chloride blank must be analyzed with each sequence and the PRO concentration shall be less than the MDL of the method.
Procedure	7.5.4 Baseline correction is allowed to correct for rises due to temperature programming. Range integration is corrected by the automatic subtraction of the baseline established by activation of a programmed run without the injection of any material. Instrument baseline must be established for every batch of samples.	9.5.4 Do not baseline subtract
Procedures	5.7 The standards are prepared at the following five different concentrations: 200 ug/ml, 100 ug/ml, 50 ug/ml, 20 ug/ml, and 5 ug/ml (per each component).	7.4.3 Suggested calibration levels are 5, 50, 150, 250, 350 and 500 ug/mL of each individual component.

TITLE: **DETERMINATION OF PETROLEUM RANGE ORGANICS (PRO) BY FLORIDA DEPARTMENT OF ENVIRONMENTAL PROTECTION METHOD # FL-PRO**

TABLE 3

METHOD ACCEPTANCE CRITERIA

	% Recovery			Precision (%RSD)		
	Water	Soil		Water	Soil	
		Soxhlet	Sonication		Soxhlet	Sonication
Matrix Spike Samples	41-101	41-224	62-204	0-20	0-25	0-25
Laboratory Control Spike Samples	55-118	63-135	63-153	0-20	0-25	0-25
Surrogates: OTP	82-142	57-115	62-109			
n-triacontane-D_{62}	70-130	70-130	70-130			

TABLE 4

PQLS

Analyte	Water ug/L	Soil mg/Kg
PRO	500	20

TITLE: **DETERMINATION OF PETROLEUM RANGE ORGANICS (PRO) BY FLORIDA DEPARTMENT OF ENVIRONMENTAL PROTECTION METHOD # FL-PRO**

FIGURE 1

EXAMPLE OF RUNLOG PAGE

Katahdin Analytical Services, Inc.
Instrument: GC12 (FID)
Amount Injected 1ul

Reviewed by/ Date:

GC Laboratory Instrument Runlog

Method (circle): EPH(MADEP) / FL PRO / TNRCC 1005

DRO/TPH - 8015Mod. / MDEP 4.1.25 / 8100Mod.

Date	Init.	Result File	Sample ID	Y/N	Method	Column	Comments
030508	KGS	CRC2029	50 ug/ml	Y	AROR0315A	289	
		30	10 ug/ml	Y			
		31	1 ug/ml	Y			
		32	ARO IND 50	Y			
		33	WG48780-1 3510	Y			conf
		34	SB0572-4	Y			10
030608		35	Mech	Y	ALIR0222A		
		36	Thermal bleed	N			
		37	PHC 50 ug/ml	Y			concentrated / new cap.
		38	ARO 50 ug/ml	Y	AROR0157A		
030708	KGR	39	Thermal bleed	N	FLP0164	289	Alimen
		40		N			
		41		Y			
		42	FLP50ug/ml	Y			H1709
		43	200	Y			H1713
		44	100	Y			H1712
		45	20	Y			H1710
		46	5	Y			H1711
		47	IND 50ug/ml	Y			H1714
		48	WG48627-1 3510	Y			
		49	-2	Y			
		50	-3	Y			
		51	SB0864-1	Y			
		52	-2	Y			
		53	Mech	Y			
		54	Thermal bleed	Y			
		55	FLP50ug/ml	Y			H1709
031008	KGR	59	TB	N	ALI60322A		
		60		Y			
		61	PHC 50ug/ml	Y			

QAGC186 0000052

TITLE: **DETERMINATION OF PETROLEUM RANGE ORGANICS (PRO) BY FLORIDA DEPARTMENT OF ENVIRONMENTAL PROTECTION METHOD # FL-PRO**

FIGURE 2

EXAMPLE OF DATA REVIEW CHECKLIST

Verbal Due Date _____ Due Date_____

Client:		Primary	Secondary
Method:		Date:	Date:
SDG No:	Level:	Initials:	Initials:
KAS No:			Approved : ☐ Yes

PRIMARY REVIEW CHECKLIST

Highlight Method / project specific information. _____

All needed forms are present . _____

Sample Data Summary Included (Level III & IV). _____

Correct Work Order Number or SDG name (all forms). _____

Correct project name and spelling (all forms). _____

Correct file numbers (all forms). _____

Analysis Date Correct. _____

Extraction Method & Analysis Method Correct. _____

Product list compared to ROAs (compounds & PQLs). _____

Chromatogram reviewed for unlabeled peaks (check product list). _____

Flagging of all ROAs correct (Florida Flagging ☐). _____

All tunes included (level IV) . _____

All log book pages included (Soil weights,TCLP & SPLP). _____

Verify quant results for CLP. _____

Update sample history files. _____

Sign & Date Manual integration (Narrate as needed). _____

Sample I.D's Truncated (NARRATE). YES ☐ Please list KAS # below :

_____

First correction ☐ → Review and replace appropriate SDS Forms ☐.

Second correction ☐ → Review and replace appropriate SDS Forms ☐.

Last saved by kasgc04/13/07

KATAHDIN ANALYTICAL SERVICES, INC.
STANDARD OPERATING PROCEDURE

SOP Number: CA-500
Revision History
Cover Page
Page 1

TITLE: PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT PESTICIDES/PCBs ANALYSIS

Prepared By: Mike Thomas Date: 7/96

Approved By:

Group Supervisor: Michael F. Thomas Date: 11/15/00

Operations Manager: [signature] Date: 10/25/00

QA Officer: Deborah J. Nadeau Date: 10.23.00

General Manager: Duncan F. Hughes Date: 11/16/00

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
01	Minor changes throughout. Clarifications to procedure section.	DN	10.23.00	10/23/00
02	removed references to medium level extraction. New logbook figures Minor changes throughout	LAD	020305 ~~02~~ LAD 020305	020305
03	updated compound list changes in wording to clarify updated logbook	LAD	04/06	04/06
04	Added definitions, added waste information, added LCSD, updated solvent exchange, updated Table 1, replaced fig. 2, added PCB cleanup to Sect. 2	LAD	09/07	09/07
05	Updated LB example. Added temp. of nitrogen water bath, lot numbers of filter paper, lot #'s of acids need to be recorded in LB. Change "N-Lo" waste to "K" waste.	LAD	07/08	07/08

TITLE: PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT PESTICIDES/PCBs ANALYSIS

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
06	Added requirement to add spike before Na_2SO_4. Changed N_2 waterbath temperature from < 37°C to < 30°C. Removed respirator references. Added KAEHS manual, Added CA-108 reference for subsampling	LAP	02/09	02/09

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT PESTICIDES/PCBs ANALYSIS**

Please acknowledge receipt of this standard operating procedure by signing and dating both of the spaces provided. Return the bottom half of this sheet to the QA Department.

I acknowledge receipt of copy ___ of document **SOP CA-500-06**, titled **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT PESTICIDES/PCBs ANALYSIS.**

Recipient: _____ Date: _____

KATAHDIN ANALYTICAL SERVICES, INC.
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy ___ of document **SOP CA-500-06**, titled **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT PESTICIDES/PCBs ANALYSIS.**

Recipient: _____ Date: _____

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT PESTICIDES/PCBs ANALYSIS**

1.0 SCOPE AND APPLICATION

The purpose of this SOP is to describe the procedures and requirements for the preparation of solid samples for pesticides/PCBs analysis in accordance with SW-846 Method 3550, current revision..

1.1 Definitions

METHOD BLANK (laboratory reagent blank): An artificial sample designed to determine if method analytes or other interferences are present in the laboratory environment, the reagents, or the apparatus. For aqueous samples, reagent water is used as a blank matrix; for soil samples, baked organic-free sand is used as a blank matrix. The blank is taken through the appropriate steps of the process.

LABORATORY CONTROL SAMPLE (LCS): A blank that has been spiked with the analyte(s) from an independent source, and is analyzed exactly like a sample. Its purpose is to determine whether the methodology is in control, and whether the laboratory is capable of making accurate and precise measurements. The matrix used should be phase matched with the samples and well characterized.

MATRIX SPIKE/MATRIX SPIKE DUPLICATE (MS/MSD): Predetermined quantities of stock solutions of certain analytes are added to a sample matrix prior to sample extraction and analysis. Samples are split into duplicates, spiked and analyzed. Percent recoveries are calculated for each of the analytes detected. The relative percent difference between the samples is calculated and used to assess analytical precision.

SURROGATES: Organic compounds which are similar to analytes of interest in chemical composition, extraction and chromatography, but which are not normally found in environmental samples. These compounds are spiked into all blanks, standards, samples and spiked samples prior to analysis. Percent recoveries are calculated for each surrogate.

1.2 Responsibilities

This method is restricted to use by, or under the supervision of, analysts experienced in the extraction of sediment/soil samples for pesticides/PCBs analysis. Each analyst must demonstrate the ability to generate acceptable results with this method.

It is the responsibility of all Katahdin personnel involved in the preparation of solid samples for pesticides/PCBs analysis to read and understand this SOP, adhere to the procedures outlined, and to properly document their data in the appropriate lab notebook. Any deviations from the test or irregularities with the samples should also

be recorded in the lab notebook and reported to the Department Manager or designated qualified data reviewer responsible for the data.

It is the responsibility of the Department Manager to oversee that the members of his/her group follow this SOP, that their work is properly documented, and to indicate periodic review of the associated logbooks.

Users of this procedure must be cognizant of inherent laboratory hazards, proper disposal procedures for contaminated materials, and appropriate segregation of hazardous wastes. Everyone involved with the procedure must be familiar with the material safety data sheets for all the materials used in this procedure. Each qualified analyst or technician must be familiar with Katahdin Analytical safety procedures.

1.3 Safety

Users of this procedure must be cognizant of inherent laboratory hazards, proper disposal procedures for contaminated materials and appropriate segregation of hazardous wastes. The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical should be treated as a potential health hazard. A reference file of material safety data sheets is available to all personnel involved in the chemical analys is. Everyone involved with the procedure must be familiar with the MSDSs for all the materials used in this procedure.

Each qualified analyst or technician must be familiar with Katahdin Analytical Environmental Health and Safety Manual including the Katahdin Hazardous Waste Plan and must follow appropriate procedures. These include the use of appropriate personal protective equipment (PPE) such as safety glasses, gloves and lab coats when working with chemicals or near an instrument and not taking food or drink into the laboratory. Each analyst should know the location of all safety equipment. Each analyst shall receive a safety orientation from their Department Manager, or designee, appropriate for the job functions they will perform.

1.4 Waste Disposal

Wastes generated during the preparation of samples must be disposed of in accordance with the procedures described in the current revision of the Katahdin Analytical Environmental Health and Safety Manual and SOP SD-903, “Sample Disposal,” current revision. Expired standards are lab packed, placed in the Katahdin hazardous waste storage area, and disposed of in accordance with this SOP.

Any methylene chloride solvent waste generated during the rinsing of glassware etc. should be disposed of in the “D” waste stream satellite accumulation area nearest

the point of generation. Acetone and hexane are considered flammable waste, and should be disposed of in the "O" waste stream satellite accumulation area nearest the point of generation. Post-extraction soil samples, used glass wool, and sodium sulfate waste should be disposed of in the soil with organics "I" waste stream satellite accumulation area nearest the point of generation. Acid waste generated during the cleanup of PCB samples should be disposed of in the "K" satellite accumulation area nearest the point of generation. Please refer to the current revision of SOP CA-107 for the location of satellite waste accumulation areas.

2.0 SUMMARY OF METHOD

Pesticides/PCBs are extracted from solid samples by sonication with a methylene chloride/acetone solution (1:1 by volume) following EPA Method 3550, current revision. The resulting extract is dried, concentrated, and solvent exchanged to hexane for analysis by GC. Sulfuric acid cleanup is performed on extracts for 8082 PCB analysis.

This SOP applies to low level extraction of pesticide/PCB pollutants from solid sample matrices.

3.0 INTERFERENCES

Solvents, reagents, glassware, and other sample preparation apparatus may yield interferences to GC analysis due to the presence of contaminants. These contaminants can lead to discrete artifacts or elevated baselines in chromatograms. Routinely, all of these materials must be demonstrated to be free from interferences under the conditions of the analysis by running reagent blanks. Interferences caused by phthalate esters can pose a major problem in pesticide analysis. Common flexible plastics contain varying amounts of phthalates that are easily extracted during laboratory operations, so cross-contamination of glassware frequently occurs when plastics are handled. Interferences from phthalates can best be minimized by avoiding the use of such plastics in the laboratory. At no time may gloves that have not been tested for phthalates or gloves known to contain phthalates be used or stored in the organic extraction lab. Whenever possible, plastic items in this lab, must be replaced with metal, teflon or other non-phthalate plastic substitute.

Special care should be taken to ensure clean glassware and apparatus are used, pre-rinsed with the appropriate solvent prior to use. Solvents should be analyzed prior to use to demonstrate that each lot is free of contaminants that may interfere with the analysis.

Interferences coextracted from the samples will vary considerably from source to source. If analysis of an extracted sample is prevented due to inteferences, further cleanup of the sample extract may be needed to minimize interferences.

4.0 APPARATUS AND MATERIALS

Prior to use, all glassware must be rinsed three times with methylene chloride.

4.1 Beakers - 400 mL

4.2 Kuderna-Danish (KD) apparatus - Concentrator tube - 10 mL
Evaporative flask - 500 mL
Snyder column - 3-ball macro

4.3 Powder funnels, 100 mm diameter, 35 mm stem

4.4 Vacuum filtration flask - 500 mL Erlenmeyer

4.5 Buchner funnel, porcelain, Coors® with 85 mm plate diameter (or equivalent)

4.6 Sonic disruptor – Misonix XL2015 (or equivalent), equipped with dual titanium horn extenders for extracting two samples at a time.

4.7 Spatula - stainless steel

4.8 Filter paper, 18.5 cm, Fisherbrand or Whatman 114V (or equivalent)

4.9 Boiling chips - 12 mesh, silicon carbide (or equivalent)

4.10 Water bath - eight position concentric ring bath or equivalent, equipped with a calibrated thermometer

4.11 Filter paper - 7.0 cm, Whatman, #4, or equivalent

4.12 Syringe - gas tight, 1.0 mL, solvent rinsed between each use

4.13 Balance – top-loading, capable of weighing to 0.1 g

4.14 Nitrogen evaporation apparatus

5.0 REAGENTS

5.1 Sodium sulfate - (ACS reagent grade) powdered, anhydrous, certified by the manufacturer/vendor as purified by heating to 400 °C prior to receipt by the laboratory. Solvent rinse immediately before use by rinsing three times with pesticide grade methylene chloride.

5.2 Sodium sulfate - (ACS reagent grade) granular, anhydrous, purified as described in section 5.1.

5.3 Methylene chloride - (pesticide grade or equivalent) purchased by lot, evaluated prior to use by concentration of 300mLs to 1.0 mL followed by GC/MS analysis.

5.4 Acetone and hexane - pesticide grade or equivalent

5.5 Organic-free sand, purified by baking at 400 °C for four hours. Method blanks serve as checks on the baked sand.

5.6 Surrogate spiking solution - Prepare a solution of decachlorbiphenyl (DCB) and tetrachloro-meta-xylene (TCMX) at a concentration of 1 ug/mL each in acetone. Store the solution at –10 to -20 °C (±2 °C) in a Teflon sealed container. Solution must be verified by GC/ECD prior to use, and must be replaced every 6 months or sooner if degradation is evident.

5.7 Pesticide Matrix spike/Lab control sample spiking solution - Prepare a spiking solution in pesticide grade methanol that contains all target analytes listed below:

Analyte	ug/mL
4,4'-DDD	0.5
4,4'-DDE	0.5
4,4'-DDT	0.5
Aldrin	0.5
alpha-BHC	0.5
beta-BHC	0.5
delta-BHC	0.5
Dieldrin	0.5
Endosulfan I	0.5
Endosulfan II	0.5
Endosulfan Sulfate	0.5
Endrin	0.5
Endrin Aldehyde	0.5
Endrin Ketone	0.5
gamma-BHC (Lindane)	0.5
Heptachlor	0.5
Heptachlor Epoxide	0.5
Methoxychlor	0.5
alpha-Chlordane	0.5
gamma-Chlordane	0.5

Store the solution at –10 to -20 °C (±2 °C) in a Teflon sealed container. Solution must be verified by GC/ECD prior to use, and must be replaced every 6 months or sooner if degradation is evident.

5.8 PCB Matrix Spike/Lab Control Sample Spiking Solution – Prepare spiking solution in pesticide grade acetone that contains PCB's Arochlor 1016 and Arochlor 1260 (1660), both at 5.0 ug/mL.

Store the solution at –10 to -20 °C (±2 °C) in a Teflon sealed container. Solution must be verified by GC/ECD prior to use, and must be replaced every 6 months or sooner if degradation is evident.

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

Soil samples are collected in glass soil jars and stored at 4°C (±2°C) until time of extraction.

Holding time for extraction of sediment/soil samples for Method 3550 is 14 days from date of sample collection, although the analyst should be aware that actual holding times employed may be project/program specific.

Store all extracts at 4°C (±2°C) in the dark in labeled Teflon-sealed containers. See SOP SD-902, "Sample Receipt and Internal Control," current revision, for storage areas and temperature maintenance procedures.

7.0 PROCEDURES

LOW LEVEL EXTRACTION OF SOIL/SEDIMENT FOR PESTICIDES/PCBs

The low level extraction procedure is designed for the preparation of soil/sediment samples that may contain analytes at levels lower than 20,000 ug/kg. The procedure involves extraction of pesticides and PCBs from an initial sample weight of 30.0 ± 0.1 g using an ultrasonic cell disruptor.

Many solid samples may need to be cleaned up to reduce matrix interferences. The cleanup procedure employed will be dependent upon the nature of the interferences and the target compounds to be analyzed, and options may include acid wash, sulfur cleanup, florisil cleanup, or gel permeation chroma tography (GPC). The Department Manager should be consulted to determine if a particular sample should be subjected to further cleanup procedures; the decision should consider sample history, sample appearance, and project/client needs. All extracts or extract splits for subsequent 8082 PCB analysis will, at a minimum, undergo acid cleanup. (Refer to SOP CA-525, current revision)

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT PESTICIDES/PCBs ANALYSIS**

7.1 Discard any excess water on the sediment sample. Mix with a stainless steel spatula to ensure homogeneity of the sample. If the sample container is full to the extent that stirring the sample is impractical, try to remove the "best representative" aliquot from the jar based on color, particle size, moisture, etc. Remove any foreign objects such as sticks, leaves, or rocks, and note actions taken in the appropriate extraction logbook. **Please refer to the current revision of Katahdin Analytical Services SOP CA-108, "Basic Laboratory Technique", for more detailed guidance on subsampling to ensure reproducibility.**

7.2 Weigh out a 30.0 $\pm$ 0.1 g portion of sample into a labeled 400 mL beaker. Record sample weight to nearest 0.1 g in appropriate extraction logbook. Refer to sections 7.6 and 7.7 for spike and surrogate addition instructions. Add between 30 g and 60 g of powdered sodium sulfate, as required, to produce a "free-flowing" mixture. The amount of sodium sulfate added will depend upon the moisture content of the sample (e.g., low moisture content will require less sodium sulfate). Mix well with a spatula. Keep the spatula in the sample beaker and cover the beaker with aluminum foil.

7.3 A method blank must be prepared with each extraction batch, not to exceed 20 client samples. To prepare method blank, weigh out one 30.0 $\pm$ 0.1 g portion of purified sand in a labeled 400 mL beaker. Refer to sections 7.6 and 7.7 for spike and surrogate addition instructions. Add 60 g sodium sulfate and mix well. (Although a "free-flowing" mixture can be achieved with less than 60 g sodium sulfate, the method blank must contain 60 g in order to evaluate the sodium sulfate as a potential source of contamination.)

7.4 A laboratory control sample (LCS) must be prepared with each extraction batch, not to exceed 20 client samples. To prepare LCS, weigh out one 30.0 $\pm$ 0.1 g portion of purified sand in a labeled 400 mL beaker. Refer to sections 7.6 and 7.7 for spike and surrogate addition instructions. Add 30 g sodium sulfate and mix well. With extraction batches prepared for combined 8081/8082 Pesticide and PCB analysis, separate Pesticide and PCB LCS's must be prepared (refer to sections 5.7 and 5.8). If an MS/MSD pair is not extracted on a particular day, an LCS/LCSD pair may be required in order to meet client-specific or program-specific requirements. This information will be disseminated from the project manager or Department Manager.

7.5 A matrix spike/matrix spike duplicate (MS/MSD) set must be prepared for every 20 samples. To prepare MS/MSD, weigh out 30.0 $\pm$ 0.1 g portions of the sample designated for MS/MSD into each of two labeled 400 mL beakers. Record sample weights to nearest 0.1 g in appropriate extraction logbook. Refer to sections 7.6 and 7.7 for spike and surrogate addition instructions. Add 30 - 60 g sodium sulfate to each to produce a free-flowing mixture, and mix well. With extraction batches prepared for combined 8081/8082 Pesticide and PCB analysis, separate Pesticide and PCB MS/MSD pairs must be prepared (refer to sections 5.7 and 5.8).

7.6 To all samples, method blank, LCS/LCSD, and MS/MSD add 1.0 mL pesticide/PCBs surrogate spiking solution using a 1.0 mL gas tight syringe. The surrogate spike should be added **prior** to the addition of the sodium sulfate. Record surrogate spike volume and identification code in extraction logbook. Thoroughly rinse syringe with solvent prior to each use.

7.7 To LCS/LCSD and MS/MSD pairs add 1.0 mL of pesticide or PCB matrix spike/LCS spiking solution using a 1.0 mL gas tight syringe. The LCS/MS spike should be added **prior** to the addition of the sodium sulfate. Record matrix spike/LCS spiking solution volume and identification code in extraction logbook. Thoroughly rinse syringe with solvent prior to spiking a different solution and when spiking is completed.

7.8 To assure optimum operation and maximum energy output, the sonicators must be tuned daily prior to extracting samples. The following tuning procedure must be performed with the sonicator probes vibrating in air.

7.8.1 Turn OUTPUT CONTROL knob counter-clockwise to zero, and turn Pulsar Duty Cycle to off (or continuous mode).

7.8.2 Press POWER SWITCH to ON (up) position. Engage the Timer Switch (W-375)

7.8.3 Press and hold down the TUNE switch.

7.8.4 Turn the Output Control Knob towards setting 10. Note the position of the needle on the % output power meter. **DO NOT exceed 70%. If you reach 70% - STOP!!** Rotate the Tuning Knob clockwise or counter-clockwise until a minimum (not maximum) reading (usually less than 20%) is obtained.

7.8.5 Turn the Output Control Knob towards setting 10. Again, note the position of the needle and do not exceed 70%. Rotate the Tuning Knob until you obtain a minimum reading of 20% or below.

7.8.6 Release the TUNE switch. **CAUTION: Do NOT touch probe. The probe is still active.**

7.8.7 Turn OUTPUT CONTROL KNOB counter-clockwise to zero (or disengage timer).

7.8.8 Confirm that the sonicators were tuned by recording the date and/or percent in the extractions logbook.

Note: If the unit will not be used immediately, please turn the power switch to off.

7.9 Prior to extracting any samples, ensure that the sonicator probes are decontaminated by rinsing with a methylene chloride wash bottle. Collect the waste in a waste beaker. It may sometimes be necessary to wipe the upper part of each probe with a methylene chloride dampened KimWipe. Repeat this decontamination step between each sample on each probe.

7.10 To the mixed and spiked blank and LCS, add 100 mL of the 1:1 methylene chloride/acetone solution and proceed with steps 7.11 through 7.14.

7.11 It may be necessary at this time to stir the sample/sodium sulfate mixture with the spatula to loosen up the mixture prior to extracting. Position beaker in the ultrasonic cell disruptor so that the bottom surface of the tip of the 3/4 inch disruptor horn is about halfway below the surface of the solvent and above the sediment layer.

7.12 Sonicate for 3 minutes with the output control knob set at 10, and mode switch on "pulsed" and % duty cycle knob set at 50%. While the mixture is sonicating, one should be able to see all, or most of the material, moving in the beaker under the influence of the energized probes. If not, stir the mixture again.

7.13 Prepare a filter flask fitted with a Buchner funnel. The Buchner funnel should contain a 7.0 cm Whatman #4 filter; prerinse flask, funnel and filter with methylene chloride and discard rinsings into solvent waste container. Decant extract into the filter flask through Buchner funnel. A vacuum pump may be used to facilitate filtration or the extract may be gravity filtered.

Note: The lot number of the filter paper must be recorded in the extraction logbook.

7.14 Repeat the extraction two additional times using 100 mL portions of 1:1 methylene chloride:acetone. Before each extraction, make certain that the sodium sulfate is free-flowing and not a consolidated mass. As required, break up large lumps with the clean spatula. Decant the extraction solvent into the Buchner funnel after each sonication. On the final sonication, pour the entire sample contents into the Buchner funnel and rinse thoroughly with 1:1 methylene chloride:acetone to complete the quantitative transfer of the extract.

CONCENTRATION OF LOW LEVEL EXTRACTS

7.15 Rinse the K-D glassware (flask, concentration tube, and Snyder column, including the ground glass joints on the flask and columns) three times with methylene chloride before assembling. Add two boiling chips to the K-D. Insert 18.5 cm filter papers into short stem powder funnels and add ~ 2 inches of sodium sulfate

crystals. Rinse the sodium sulfate in the assembled funnels with ~20-30 mLs of methylene chloride. Place the assembled K-D's under the funnels.

Note: The lot number of the filter paper must be recorded in the extraction logbook.

7.16 For a solvent exchange, add approximately 50 mL Hexane to funnel and let drain through. Since methylene chloride has a lower boiling point than Hexane, this will result in a final extract in hexane only.

7.17 Transfer the extracts to the K-D concentrator setups through the sodium sulfate in the funnels. This is the drying step, which is required to remove residual water from the extracts. Any large water layers must be removed by other means, prior to pouring through the sodium sulfate. After pouring all of the extract volume through the sodium sulfate, rinse the extract flask three times with ~ 2 – 3 mLs of methylene chloride. Add the rinsings through the sodium sulfate to complete a quantitative transfer. Rinse the sodium sulfate with ~ 15 mLs of methylene chloride and allow to drain.

7.18 If samples are to be GPC'd, refer to the current revision of Katahdin SOP CA-513, Extract Cleanup Using Gel Permeation Chromatography, for appropriate concentrating procedures.

7.19 If samples are not to be GPC'd follow Steps 7.19 through 7.23 to concentrate extracts to final volume of 10.0 mLs. Otherwise proceed to GPC cleanup procedure as described in the current revision of Katahdin SOP CA-513.

7.20 Transfer the labels from the extract flasks to the K-Ds. Remove the funnel and attach a 3-ball macro Snyder column. Pre-wet the Snyder column with 1 mL of methylene chloride.

7.21 Place the K-D in a hot water bath (75-85°C). Gently swirl K-D in the water until boiling begins. At the proper distillation rate, the Snyder balls should chatter but the chambers should not flood with condensed solvent. The K-D should be kept in a vertical orientation while on the bath. When the apparent volume in the concentrator tube reaches ≈ 6 mL, remove the K-D from the water bath. Allow the K-D to cool for 10 minutes. Rinse the Snyder column lower joint with ≈ 1 mL of methylene chloride. Remove the Snyder column. Wipe off any water from the neck above the lower joint of the flask. Separate the K-D flask from the concentrator tube, rinsing the ground glass joint with ≈ 1 mL methylene chloride.

7.22 Reduce the extract in the concentrator tube to approximately 10 mL using the nitrogen blow-down apparatus. The bath temperature must be no higher than 30° C. Turn the gas to 3 psi. Be careful not to splash the extract out of the tube. During concentration on the N-evap, the internal wall of the concentrator tube and the N-

evap sparging pipet must be rinsed down at least once or twice with ≈1 mL of methylene chloride. The solvent level in the concentrator tube must be positioned below the level of the water bath as much as possible to prevent water from condensing into the sample extract. As the extract volume is reduced, lower the N_2 sparging pipet closer to the surface of the extract to expedite the concentration. Note any problems or extract losses, if they occur, in the extractions logbook.

Note: The temperature of the water in the nitrogen evaporation water bath must be recorded in the logbook.

7.23 Complete quantitative transfer of the extract to a vial by using hexane. Adjust the volume of the hexane extract to 10 mL in either a 12 or 16 mL vial using the appropriate "reference vial" for volume comparison.

7.24 Transfer the sample label from the concentrator tubes to the vials. Store extract vials at a temperature of 4 ± 2 °C until ready for analysis. Indicate in the extractions logbook the box number and "tray location" of the individual extract vials.

7.25 All sample extracts for 8082 PCB analysis must undergo a sulfuric acid wash (cleanup) prior to analysis. All sample extracts for 8081 pesticide analysis do not undergo further cleanup unless requested by the client. All sample extracts for combined 8081/8082 analyses must be split. One portion must be acid cleaned for 8082 analysis. The associated method blank must be split and acid-cleaned in the same fashion. Prior to splitting, contents of vial must be shaken well. PCB LCSs and matrix spikes are acid cleaned also. Pesticide LCSs and matrix spikes are not subjected to further cleanup. Please refer to Katahdin SOP CA525 (current revision), Extract Cleanup Using Sulfuric Acid, for further instructions.

Note: The lot number of the acid used in PCB cleanup must be recorded in the extraction logbook.

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

Each extractions analyst must demonstrate proficiency in performing the extractions that prepare samples for analysis. Demonstration consists of preparation (extraction), by the analyst, of at least four aliquots which are then analyzed according to the analytical method in question. These QC samples must meet all quality control acceptance limits. Demonstration must be documented by use of a form which summarizes the results of the analysis of these aliquots, calculated percent recoveries, and standard deviation. Demonstration of proficiency must be done one time per analyst initially and then annually thereafter. Refer to SOPs QA-805 and QA-807, current revision.

If, upon analysis of the extracted samples, it is discovered that quality control acceptance criteria have not been met, all associated samples must be evaluated against all the QC. In some cases data may be reported, perhaps with narration, while in other cases, other corrective action may be taken. The corrective actions may include re-extraction of the samples associated with the quality control sample that did not meet acceptance criteria, or may include making n ew reagents and standards if the standardization is suspect. These decisions are based on holding time considerations, client and project specific Data Quality Objectives and on review of chromatograms. The supervisor, Operations Manager, and/or Quality Assurance Officer may be consulted to evaluate data. Some samples may not be able to be reanalyzed within hold time. In these cases "qualified" data with narration may be advisable after consultation with the client.

Much of the work performed at the lab is analyzed in accordance with specific QC requirements spelled out in a project specific Quality Assurance Project Plan (QAPP) or in a program specific Quality Systems Manual (QSM). The reporting limits, acceptance criteria and/or corrective actions may be different than those specified in this SOP. In these cases the appropriate information will be communicated to the Department Manager and/or senior chemists before initiation of the analyses so that specific product codes can be produced for the project. In addition, the work order notes for each project will describe the specific QAPP or QSM to be followed.

8.1 A method blank must be extracted for <u>each</u> and <u>every</u> item listed below:

- Each extraction method
- Every extraction batch of twenty or fewer samples
- Each analysis (pesticides and/or PCBs)

8.2 A laboratory control sample (LCS) is required for <u>each</u> and <u>every</u> item listed below:

- Each extraction method
- Every extraction batch of twenty or fewer samples
- Each analysis (pesticides and/or PCBs)

Refer to the current revision of the applicable Katahdin SOP for analysis of Pesticides and PCBs for quality control acceptance criteria.

9.0 METHOD PERFORMANCE

Refer to the applicable analytical SOP.

10.0 APPLICABLE DOCUMENTS/REFERENCES

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT PESTICIDES/PCBs ANALYSIS**

Test Methods for Evaluating Solid Waste - Physical/Chemical Methods, Method 3550C, USEPA SW-846, Third Edition, Final Update IV, February 2007.

Table 1 Summary of Method Modifications
Figure 1 Example of Pest./PCB Soil Sample Prep Logbook Page

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT PESTICIDES/PCBs ANALYSIS**

TABLE 1

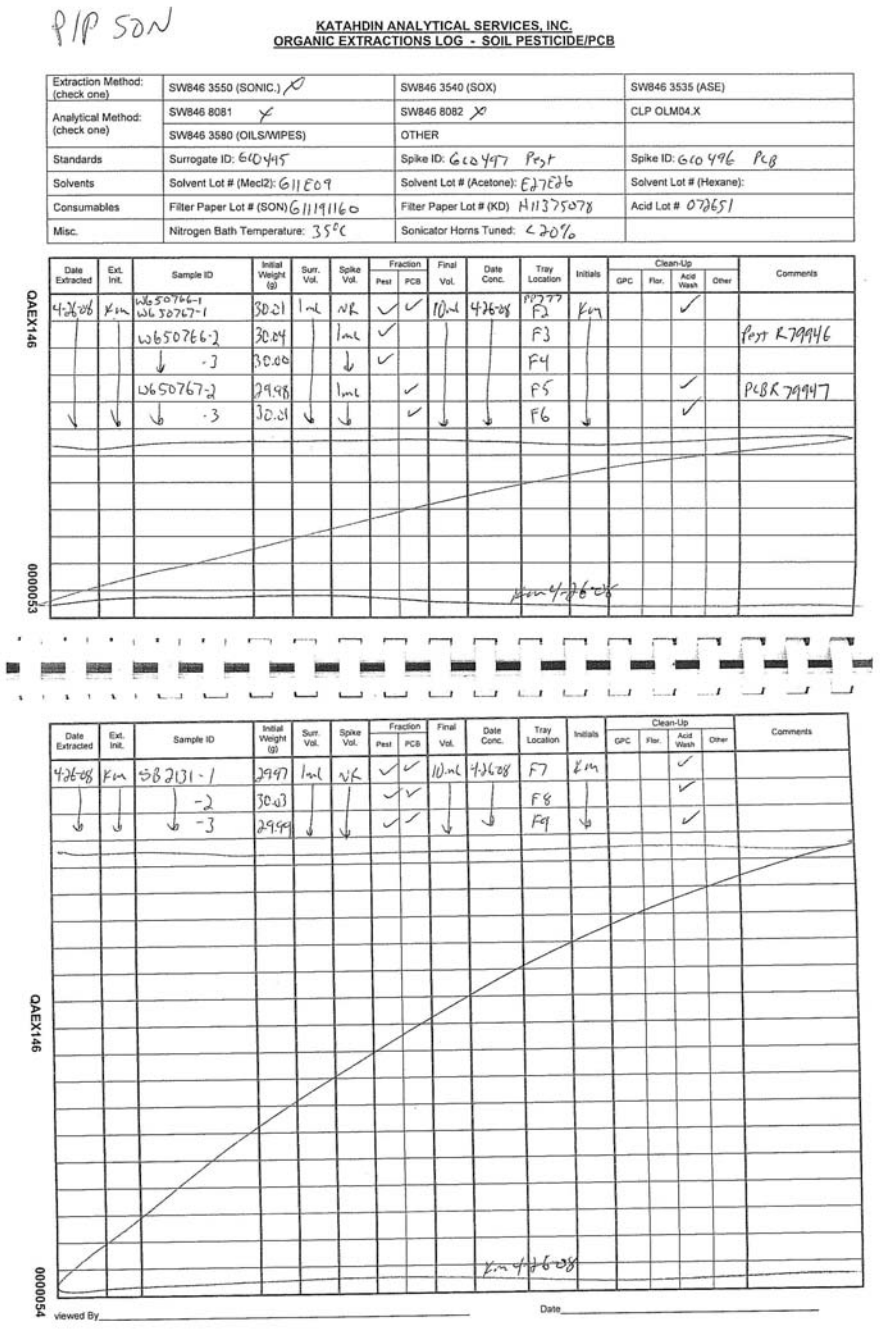
SUMMARY OF METHOD MODIFICATIONS

TOPIC	KATAHDIN SOP CA-500-06	METHOD 3550, current revision
Apparatus/Materials	1. short stem funnels	1. drying columns
Reagents		
Sample preservation/ handling		
Procedures	1. extract dried using Na_2SO_4 in short stem funnels 2. place sonicator horns ½ way between the surface of the solvent and the sediment layer 3. no apparatus height specification for concentration on water bath 4. water bath at 75-85 deg C 5. sample removed from water bath when volume reaches ~6 mL 6. Solvent exchange to hexane is performed using K-D apparatus with addition of approximately 50 mL hexane at the start of concentration process	1. extract dried using Na_2SO_4 in drying columns 2. place sonicator horns ½ inch below the solvent surface but above sediment layer 3. partially immerse concentrator tube in water and lower apparatus to complete concentration in 10-15min 4. water bath at 80-90 deg C 5. sample removed from water bath when volume reaches 1-2 mL 6. Solvent exchange to hexane is performed using K-D apparatus with addition of approximately 50 mL hexane after concentrating methylene chloride extract to 1 mL
QC - Spikes	Refer to analytical SOP	
QC - LCS		
QC - Accuracy/Precision		

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT PESTICIDES/PCBs ANALYSIS**

FIGURE 1

EXAMPLE OF LOGBOOK PAGE



P/P SON

KATAHDIN ANALYTICAL SERVICES, INC.
ORGANIC EXTRACTIONS LOG - SOIL PESTICIDE/PCB

Extraction Method: (check one)	SW846 3550 (SONIC.) ✓	SW846 3540 (SOX)	SW846 3535 (ASE)
Analytical Method: (check one)	SW846 8081 ✓	SW846 8082 ✓	CLP OLM04.X
	SW846 3580 (OILS/WIPES)	OTHER	
Standards	Surrogate ID: 6C0495	Spike ID: 6C0497 Pest	Spike ID: 6C0496 PCB
Solvents	Solvent Lot # (MeCl2): 611E09	Solvent Lot # (Acetone): E27E86	Solvent Lot # (Hexane):
Consumables	Filter Paper Lot # (SON) 611191160	Filter Paper Lot # (KD) H113750781	Acid Lot # 072651
Misc.	Nitrogen Bath Temperature: 35°C	Sonicator Horns Tuned: < 20%	

Date Extracted	Ext. Init.	Sample ID	Initial Weight (g)	Surr. Vol.	Spike Vol.	Fraction Pest	Fraction PCB	Final Vol.	Date Conc.	Tray Location	Initials	Clean-Up GPC	Clean-Up Flor.	Clean-Up Acid Wash	Clean-Up Other	Comments
4-26-08	KM	W650766-1	30.01	1mL	NA	✓	✓	10mL	4/26/08	F2	KM			✓		
		W650766-2	30.04	1mL		✓				F3						Pest K79946
		↓ -3	30.00	↓		✓				F4						
		W650767-2	29.98	1mL			✓			F5				✓		PCBK79947
		↓ -3	30.01	↓			✓			F6				✓		

QAEX146 0000053

Date Extracted	Ext. Init.	Sample ID	Initial Weight (g)	Surr. Vol.	Spike Vol.	Fraction Pest	Fraction PCB	Final Vol.	Date Conc.	Tray Location	Initials	Clean-Up GPC	Clean-Up Flor.	Clean-Up Acid Wash	Clean-Up Other	Comments
4-26-08	KM	SB2131-1	29.97	1mL	NA	✓	✓	10mL	4/26/08	F7	KM			✓		
		↓ -2	30.03			✓	✓			F8				✓		
		↓ -3	29.99	↓	↓	✓	✓	↓	↓	F9	↓			✓		

QAEX146 0000054

Viewed By_____ Date_____

TITLE: PREPARATION OF AQUEOUS SAMPLES FOR EXTRACTABLE SEMIVOLATILE ANALYSIS

Prepared By: Michael Thomas Date: 07-24-00

Approved By:

Department Manager: [signature] Date: 6-23-06

Operations Manager: Deborah J. Nadeau Date: 6.23.06

QA Officer: Susie Dimond Date: 6-23-06

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
03	Changes to sect. 5.5 & Figures 3 & 4 to reflect current spike solutions and concentrations Replaced cover page. Original cover page filed with SOP CA502-02	LAD	04/06	04/06
04	Added definitions, added waste information added LCSD, added SIM LCS/D, MS/D, updated Table 1, added use of narrow range pH paper. Minor changes throughout to reflect current practice.	LAD	09/07	09/07
05	Removed MS/MSD 14 day requirement. Changed CLLE extraction time to 18 → 24 hours. Added information on determining initial sample volume. Added extracted sample disposal. Removed all references to method 625.	LAD	09/08	09/08
06	Added to check pH after B/N CLLE extraction to ensure pH ≥ 11. If not add more NaOH and continue extracting. Added information for initial volume determination. Added Reference to CA-109. Updated logbook example. Added if extract goes dry - re extract.	LAD	10/09	10/09

TITLE: PREPARATION OF AQUEOUS SAMPLES FOR EXTRACTABLE SEMIVOLATILE ANALYSIS

Revision History (cont.):

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date

TITLE: PREPARATION OF AQUEOUS SAMPLES FOR EXTRACTABLE SEMIVOLATILE ANALYSIS

Please acknowledge receipt of this standard operating procedure by signing and dating both of the spaces provided. Return the bottom half of this sheet to the QA Department.

I acknowledge receipt of copy ___ of document **SOP CA-502-06**, titled **PREPARATION OF AQUEOUS SAMPLES FOR EXTRACTABLE SEMIVOLATILE ANALYSIS**.

Recipient: _____ Date: _____

KATAHDIN ANALYTICAL SERVICES, INC.
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy ___ of document **SOP CA-502-06**, titled **PREPARATION OF AQUEOUS SAMPLES FOR EXTRACTABLE SEMIVOLATILE ANALYSIS**.

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TITLE: PREPARATION OF AQUEOUS SAMPLES FOR EXTRACTABLE SEMIVOLATILE ANALYSIS

1.0 SCOPE AND APPLICATION

The purpose of this SOP is to describe procedures utilized by Katahdin Analytical personnel in the preparation of all non-CLP aqueous samples for analysis of extractable semivolatile organic compounds.

The goal of this procedure is to ensure uniformity involving the preparation of samples for subsequent SVOA analysis by GC/MS. This SOP is applicable to EPA Methods 3510 (modified separatory funnel extraction) and 3520 (continuous liquid-liquid extraction), current revisions.

1.1 Definitions

METHOD BLANK (laboratory reagent blank): An artificial sample designed to determine if method analytes or other interferences are present in the laboratory environment, the reagents, or the apparatus. For aqueous samples, reagent water is used as a blank matrix; for soil samples, baked organic-free sand is used as a blank matrix. The blank is taken through the appropriate steps of the process.

LABORATORY CONTROL SAMPLE (LCS): A blank that has been spiked with the analyte(s) from an independent source, and is analyzed exactly like a sample. Its purpose is to determine whether the methodology is in control, and whether the laboratory is capable of making accurate and precise measurements. The matrix used should be phase matched with the samples and well characterized.

MATRIX SPIKE/MATRIX SPIKE DUPLICATE (MS/MSD): Predetermined quantities of stock solutions of certain analytes are added to a sample matrix prior to sample extraction and analysis. Samples are split into duplicates, spiked and analyzed. Percent recoveries are calculated for each of the analytes detected. The relative percent difference between the samples is calculated and used to assess analytical precision.

SURROGATES: Organic compounds which are similar to analytes of interest in chemical composition, extraction and chromatography, but which are not normally found in environmental samples. These compounds are spiked into all blanks, standards, samples and spiked samples prior to analysis. Percent recoveries are calculated for each surrogate.

1.2 Responsibilities

This method is restricted to use by, or under the supervision of analysts experienced in the extraction of samples for semivolatile analysis. Each analyst must demonstrate and document their ability to generate acceptable results with this method. Refer to Katahdin SOP QA-805, current revision, “Personnel Training & Documentation of Capability”.

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It is the responsibility of all Katahdin technical personnel involved in the extraction of samples for semivolatile analysis to read and understand this SOP, to adhere to the procedures outlined, and to properly document their data in the appropriate lab notebook. Any deviations from the test or irregularities with the samples should also be recorded in the lab notebook and reported to the Department Manager or designated qualified data reviewer responsible for this data.

It is the responsibility of the Department Manager to oversee that members of their department follow this SOP, to ensure that their work is properly documented and to initiate periodic review of the associated logbooks.

1.3 Safety

Users of this procedure must be cognizant of inherent laboratory hazards, proper disposal procedures for contaminated materials and appropriate segregation of hazardous wastes. The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical should be treated as a potential health hazard. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Everyone involved with the procedure must be familiar with the MSDS's for all the materials used in this procedure.

Each qualified analyst or technician must be familiar with Katahdin Analytical Health and Safety Manual including the Katahdin Hazardous Waste Management Plan and must follow appropriate procedures. These include the use of appropriate personal protective equipment (PPE) such as safety glasses, gloves and lab coats when working with chemicals or near an instrument and not taking food or drink into the laboratory. Each analyst should know the location of all safety equipment. Each analyst shall receive a safety orientation from their Department Manager, or designee, appropriate for the job functions they will perform.

1.4 Pollution Prevention/Waste Disposal

Whenever possible, laboratory personnel should use pollution prevention techniques to address their waste generation. Refer to the current revision of the Katahdin Hazardous Waste Management Plan for further details on pollution prevention techniques.

Wastes generated during the preparation of samples must be disposed of in accordance with the Katahdin Hazardous Waste Management Plan and Safety Manual and SOP SD-903, "Sample Disposal," current revision. Expired standards are lab packed, placed in the Katahdin hazardous waste storage area, and disposed of in accordance with this SOP.

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Any methylene chloride solvent waste generated during the rinsing of glassware, disassembly of CLLEs after extraction, etc. should be disposed of in the "D" waste stream satellite accumulation area nearest the point of generation. Acetone and methanol are considered flammable waste, and should be disposed of in the "O" waste stream satellite accumulation area nearest the point of generation. Post-extraction aqueous samples are considered either N-Hi or N-Low waste and should be disposed of in the corresponding satellite waste accumulation area nearest the point of generation. Sodium sulfate used for sample drying should be disposed of in the soil with organics "I" waste stream satellite accumulation area nearest the point of generation. Please refer to the current revision of SOP CA-107 for the location of satellite waste accumulation areas.

2.0 SUMMARY OF METHOD

For aqueous samples extracted by CLLE, a one liter aliquot of sample is adjusted to pH $\leq$ 2 and extracted with methylene chloride using a continuous liquid-liquid extractor. The pH is then adjusted to pH $\geq$ 11 and the sample is extracted again with methylene chloride. A modified separatory funnel extraction may also be used. If this procedure is used, the sample aliquot is first adjusted to pH $\geq$ 11 and then to pH $\leq$ 2. The methylene chloride extract is dried and concentrated to a volume of 1.0 mL.

3.0 INTERFERENCES

Solvents, reagents, glassware, and other sample preparation apparatus may yield interferences to GC/MS analysis due to the presence of contaminants. These contaminants can lead to discrete artifacts or elevated baselines in the total ion current profiles (TICPs). Routinely, all of these materials must be demonstrated to be free from interferences under the conditions of the analysis by running reagent blanks. Interferences caused by phthalate esters can pose a major problem in semivolatile analysis. Common flexible plastics contain varying amounts of phthalates that are easily extracted during laboratory operations, so cross-contamination of glassware frequently occurs when plastics are handled. Interferences from phthalates can best be minimized by avoiding the use of such plastics in the laboratory. At no time may gloves that have not been tested for phthalates or gloves known to contain phthalates be used or stored in the organic extraction lab. Additionally, whenever possible plastic items in this lab must be replaced with metal or teflon or other non-phthalate plastic substitute.

Special care should be taken to ensure that clean glassware and apparatus are used and pre-rinsed with the appropriate solvent prior to use. Solvents should be analyzed prior to use to demonstrate that each lot is free of contaminants that may interfere with the analysis.

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Interferences coextracted from the samples will vary considerably from source to source. If analysis of an extracted sample is prevented due to inteferences, further cleanup of the sample extract may be needed to minimize interferences.

4.0 APPARATUS AND MATERIALS

Brand names and catalog numbers are included for illustration purposes only.

4.1 Continuous liquid-liquid extractors - including body, 500 mL round bottom flask and Alhin condensers and equipped with Teflon or glass connecting joints requiring no lubrication (Hershberg-Wolf Extractor, Ace Glass Company, Vineland, NJ, P/N 6841-10 or equivalent).

4.2 Glass powder funnels.

4.3 Fluted filter paper, 18.5cm diameter.

4.4 Concentrator tube - Kuderna-Danish, 10 mL, graduated (Kontes K-570050-1025 or equivalent). Calibration must be checked at the volumes employed in the test.

4.5 Evaporation flask - Kuderna-Danish, 500 mL (Kontes K-570001-0500 or equivalent). Attach to concentrator tube with neck clips.

4.6 Snyder column - Kuderna-Danish, three- or four-ball macro (Kontes K-503000-0121 or equivalent).

4.7 Syringe - gas tight, 1.0 mL, solvent rinsed between each use.

4.8 Vials - Glass, 1.8 mL capacity, with polytetrafluoroethylene (PTFE)-lined screw top and 12 mL with Teflon-lined caps.

4.9 2 L separatory funnel, equipped with Teflon stopper and stopcock; Nalgene Teflon FEP separatory funnels may also be used.

4.10 Organic Free Boiling Chips - approximately 10/40 mesh, Teflon or silicon carbide (or equivalent). Cleaned by Soxhlet for 18 hours.

4.11 Water bath - heated, with concentric ring cover, capable of temperature control (± 20°C). The bath should be used in a hood.

4.12 Nitrogen evaporation apparatus.

4.13 Wide range pH test strips, pH 0-14, Whatman CF Type.

4.14 Glass rods for stirring samples.

4.15 Amber bottles or other appropriate containers for collection of extracts from separatory funnel extraction.

4.16 5 ¾" Pasteur pipets.

4.17 Narrow range pH test strips, pH 0 to 2.5 pH, EMD ColorpHast or equivalent.

4.18 Narrow range pH test strips, pH 11 to 13 pH, EMD ColorpHast or equivalent.

5.0 REAGENTS

All reagent and solvent lots must be checked for possible contamination. Refer to the current version of Katahdin SOP CA-105, Reagent and Solvent Handling, for further details. The extraction staff is responsible for submitting samples to the GC or GC/MS sections for appropriate analysis. All information concerning preparation of the reagent/solvent lot sample will be recorded in the Organic Extraction Log (Figure 1) and acceptance or rejection of these lots must be recorded in the solvent/reagent lot check logbook (Figure 2). All reagents and solvents must be free (<PQL) of any target compounds.

5.1 Laboratory Reagent Grade Water - defined as water in which an interferent is not observed at or above the PQL of each parameter of interest. Deionized water filtered through activated charcoal.

5.2 Sodium sulfate - granular. Bake at 400°C for 4 hours (may be done by vendor). Purify by rinsing three times with pesticide grade methylene chloride. Allow residual methylene chloride to evaporate before each use. Cool in a desiccator and store in a glass bottle with a Teflon-lined cap.

5.3 Sulfuric acid solution (1:1 H_2SO_4 : H_2O) - slowly add 500 mL of H_2SO_4 (sp gr 1.84) to 500 mL reagent water.

5.4 Acetone, methanol, methylene chloride - pesticide residue analysis grade or equivalent.

5.5 Standard Preparation - For all standard preparations, see current revision of the following Katahdin Analytical SOPs:

- "Standards Preparation, Documentation and Traceability", (CA-106, current revision)
- "Balance Calibration," (CA-102, current revision)

5.5.1. Base/Neutral and Acid (SVOA) Surrogate Spiking Solution - Surrogate standards are added to all samples and calibration solutions. Prepare a surrogate standard spiking solution that contains the following compounds at the indicated concentrations in acetone.

Compound	Conc.
phenol-$_{d6}$	100 ug/mL
2,4,6-tribromophenol	100 ug/mL
2-fluorophenol	100 ug/mL
nitrobenzene-$_{d5}$	50 ug/mL
p-terphenyl-$_{d14}$	50 ug/mL
2-fluorobiphenyl	50 ug/mL

Store the spiking solution at -10°C to -20°C in Teflon-sealed containers. These solutions must be replaced after six months, or sooner if comparison with quality control check samples indicates a problem. If reanalysis of a method blank still indicates surrogates out of criteria, a new surrogate solution must be used.

5.5.2 SIM Surrogate Spiking Solution- Surrogate Standards are added to all samples and calibration solutions. Prepare a surrogate solution that contains the following compounds at a concentration of 2 ug/mL in acetone.

Compound	Conc. ug/mL
Fluorene-d10	2.0 ug/mL
2-Methylnaphthalene-d10	2.0 ug/mL
Pyrene-d10.	2.0 ug/mL
2,4-Dibromophenol	2.0 ug/mL

Store the spiking solution at -10°C to -20°C in Teflon-sealed containers. These solutions must be replaced after six months, or sooner if comparison with quality control check samples indicates a problem. If reanalysis of a method blank still indicates surrogates out of criteria, a new surrogate solution must be used.

5.5.3 SVOA Matrix Spike/Lab Control Samples Spiking Solution - the matrix spike/LCS solution consists of the compounds listed in Figure 3.
Prepare a spiking solution that contains each of the base/neutral compounds listed in Figure 3 at 50 ug/mL in methanol and the acid compounds at 100 ug/mL in methanol. Matrix spike/LCS standards are stored in the freezer (-10°C to -20°C) located in the storage area.

5.5.4 Base/Neutral (SIM) Matrix Spike/ Lab Control Sample Spike Solution for SIM-SVOA. Prepare a spiking solution in methanol that contains the compounds listed in Figure 3 at a concentration of 2 ug/mL for base/neutral.

Take out 1.0 mL of Base/Neutral and Acid Matrix Spike/Lab Control Spiking Solution for SVOA and dilute it to 25.0 mL of methanol. Store the solution Spiking at -10°C to -20°C in Teflon-sealed containers. These solutions must be replaced after six months, or sooner if comparison with quality control check samples indicates a problem.

5.5.5 Base/Neutral and Acid (SVOA) Appendix IX Lab Control Sample / Matrix Spike Spiking Solution – Prepare a spiking solution in methanol that contains the compounds listed in Figure 4 at concentrations of 100 ug/ml. Store the spiking solution at -10°C to -20°C in Teflon-sealed containers. These solutions must be replaced after six months, or sooner if comparison with quality control check samples indicates a problem.

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

Continuous liquid-liquid (Method 3520) and/or separatory funnel (Method 3510) extractions for semivolatiles must be started within seven days of date of sample collection, although the analyst should be aware that actual holding times employed may be project/program specific. If sampling date is unknown, the hold time is counted from one day prior to date received.

7.0 PROCEDURES

The following information must be recorded in the extraction logbook.

- Extraction method
- Surrogate and spike IDs
- Lot numbers of all solvents, acids and bases, sodium sulfate, filter paper
- Nitrogen evaporation water bath temperature
- Sample pH if appicable
- Extraction and Concentration dates
- Extraction and Concentration analyst
- Sample ID or QC sample ID
- Initial and final volumes or weight
- Surrogate and spike amounts
- Any sample cleanup preformed
- Final extract tray location
- Any comments regarding the sample extraction (ie. Emulsion)
- Prep batch start time and end time
- CLLE start time and end time
- Lot number of the vials the concentrated extracts are stored in.

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The internal chain-of-custody must be signed when removing and replacing samples in storage locations.

7.1 CONTINUOUS LIQUID-LIQUID EXTRACTION (Method 3520)

7.1.1 Set up the CLLE apparatus. All glassware should be pre-rinsed three times with methylene chloride in order to eliminate any contamination factors.

7.1.2 Add approximately 500 - 600 mL of methylene chloride to the CLLE body. Label each flask with the following: sample number (or QC identification number), analyte (SVOA), extraction method (CLLE), and extraction date.

7.1.3 A method blank and a laboratory control sample (LCS) must be prepared for each daily extraction batch of twenty samples or fewer (if a work order consists of more than twenty samples, a new batch must be started on a separate page with its own method blank and LCS). To prepare method blank and LCS, add 1 L reagent water to a CLLE body. Be sure that no water leaks into the round bottom flask. If combined SIM-SVOA analysis is requested, a separate LCS must be prepared for each analysis. This blank and LCS are carried through the entire extraction and analytical procedure.

7.1.4 Mark the sample level (meniscus) on the sample bottle with a wax crayon so that the volume can be measured (this may be done prior to removal from the walk-in cooler). Transfer the sample to a CLLE body, being sure that no water leaks into the round bottom flask.

7.1.5 If the batch requires a MS/MSD, transfer two 1 L portions of the sample selected/designated for MS/MSD to CLLE bodies for preparation of a matrix spike/matrix spike duplicate if required. If combined SIM-SVOA analysis is requested, a separate MS/MSD must be prepared for each analysis. If extra MS/MSD aliquots of sample are unavailable a laboratory control sample duplicate (LCSD) may be substituted.

7.1.6 Check the pH of each sample with wide range pH paper by removing a couple of sample drops with a clean disposable pipet or on the tip of a stirring rod. Adjust the pH of the samples (including method blank, LCS/LCSD, and MS/MSD) to $\leq$ pH 2 with 1:1 H_2SO_4 after addition of surrogates and spikes and prior to attaching Allihn condensers (Step 7.1.11). Stir with a glass stirring rod and check pH by tapping the glassrod onto wide range pH paper. The pH must be ≤ 2. If the pH test strip does not clearly indicate the pH is less than 2, narrow range pH paper must be used.

7.1.7 For each sample, rinse the original sample container with approximately 30 mL of methylene chloride. Add this rinse to the CLLE body.

7.1.8 Determine the initial volume of the samples by comparing the grease marking where the sample meniscus was to the reference bottle located in the lab. Record the volume and any notable characteristics (e.g. color, presence of sediment, or odor) in the extraction logbook.

7.1.9 To all samples, method blank, LCS/LCSD, and MS/MSD add 1.0 mL base/neutral and acid (SVOA) surrogate spiking solution using a 1.0 mL or 2.5 mL gas tight syringe. Record surrogate spike volume and identification code in extraction logbook. Thoroughly rinse syringe with acetone before and after each use.

7.1.9.1 If the request is for SVOA, use the SVOA Surrogate Solution (sect. 5.5.1).

7.1.9.2 If the request is for SIM, use the SIM surrogate solution (sect. 5.5.2).

7.1.9.3 If the request is for SIM-SVOA, use the SIM surrogate solution as well as SVOA surrogate solution. NOTE: Separate LCS/LCSD and/or MS/MSD are needed for each analysis and should be spiked with the appropriate surrogate.

7.1.10 To LCS/LCSD and MS/MSD add 1.0 mL base/neutral and acid (SVOA) matrix spike/LCS spiking solution using a 1.0 mL or 2.5 mL gas tight syringe. Record matrix spike/LCS spiking solution volume and identification code in extraction logbook. Thoroughly rinse syringe with methanol before and after each use.

7.1.10.1 If the request is for SVOA -
add 1.0 mL of SVOA Matrix Spike/Lab Control Samples Spiking Solution (sect 5.5.3).

7.1.10.2 If the request is for SIM -
add 1.0 mL of SVOA Matrix Spike/Lab Control Samples Spiking Solution (sect 5.5.3) and
add 1.0 mL of Base/Neutral (SIM) Matrix Spike/ Lab Control Sample Spike Solution (sect 5.5.4).

7.1.10.3 If the request is for SVOA Appendix IX, use the SVOA Appendix IX Spiking solution as well as the SVOA spiking solution -
add 1.0 mL of SVOA Matrix Spike/Lab Control Samples Spiking Solution (sect 5.5.3) and
add 1.0 mL of Base/Neutral and Acid (SVOA) Appendix IX Lab Control Sample / Matrix Spike Spiking Solution (sect 5.5.5).

7.1.11 Attach cooling water Allihn condensers, after first rinsing each 45/50 joint with methylene chloride. Turn on the heating mantles and allow the samples to extract for 18 to 24 hours. Turn off the mantles and let samples cool.

7.1.12 Detach condensers and verify that the pH is still $\leq$ 2 in the same manner mentioned in 7.1.6. If the pH has changed, more acid should be added to make the pH $\leq$ 2 and the sample extracted for several more hours.

7.1.13 Upon completion of acid extraction, allow the sample to cool. Detach condensers and add enough 10N NaOH to adjust the pH to $\geq$ 11 with stirring. Use glass stirring rods to stir and check the pH of each sample in the same manner mentioned in 7.1.6.

7.1.14 Re-attach Allihn condensers, turn on heating mantles, and allow samples to extract for 18 to 24 hours. Turn off mantles and allow samples to cool.

7.1.15 Detach condensers and verify that the pH is still $\geq$ 11 in the same manner mentioned in 7.1.6. If the pH has changed, more NaOH should be added to make the pH $\geq$ 11 and the sample extracted for several more hours.

7.1.16 Once samples are cool to the touch, the CLLE apparatus can be disassembled. The round bottom flask is removed, covered foil and placed in the interim extract refrigerator. The remaining sample in the CLLE body is poured in the "N-Hi" satellite.

Proceed to Step 7.3 for sample extract concentration procedures.

7.2 SEPARATORY FUNNEL EXTRACTION (Modified Method 3510)

If an emulsion prevents acceptable recovery or client history indicates samples may demonstrate matrix interference, then samples should be extracted by continuous liquid-liquid extraction (CLLE).

7.2.1 Rinse all glassware, including teflon separatory funnels, three times with methylene chloride prior to use.

7.2.2 Label 2 L separatory funnels and amber collection bottles clearly. Each label should include: sample number (or QC indicator number), analyte (SVOA), matrix (Aq), extraction date.

7.2.3 A method blank and a laboratory control sample (LCS) must be prepared for every 20 samples or with each extraction batch, whichever is more frequent. To prepare method blank and LCS, add 1 L reagent water to a separatory funnel. If combined SIM-SVOA analysis is requested, a separate LCS must

be prepared for each analysis. This blank and LCS are carried through the entire extraction and analytical procedure.

7.2.4 Measure the initial volume by comparing the meniscus of the sample with the reference bottle of the same bottle type. Please refer to SOP CA-108, "Basic Laboratory Technique", for the reference bottle verification procedure. Record the volume and any notable characteristics (e.g. color, presence of sediment, or odor) in the extraction logbook.

7.2.5 If the batch requires a MS/MSD, transfer two 1 L portions of the sample selected/designated for MS/MSD to separatory funnels for preparation of a matrix spike/matrix spike duplicate if required. If combined SIM-SVOA analysis is requested, a separate MS/MSD must be prepared for each analysis. If extra MS/MSD aliquots of sample are unavailable, a laboratory control sample duplicate (LCSD) may be substituted.

7.2.6 To all samples, method blank, LCS/LCSD, and MS/MSD add 1.0 mL base/neutral and acid (SVOA) surrogate spiking solution using a 1.0 mL or 2.5 mL gas tight syringe. Record surrogate spike volume and identification code in extraction logbook. Thoroughly rinse syringe with acetone before and after each use.

7.2.6.1 If the request is for SVOA, use the SVOA Surrogate Solution.

7.2.6.2 If the request is for SIM, use the SIM Surrogate Solution.

7.2.6.3 If the request is for SIM-SVOA, use the SIM surrogate solution as well as SVOA surrogate solution. NOTE: Separate LCS/LCSD and/or MS/MSD are needed for each analysis and should be spiked with the appropriate surrogate.

7.2.7 To LCS/LCSD and MS/MSD add 1.0 mL base/neutral and acid (SVOA) matrix spike/LCS spiking solution using a 1.0 mL or 2.5 mL gas tight syringe. Record matrix spike/LCS spiking solution volume and identification code in the extraction logbook. Thoroughly rinse syringe with methanol before and after each use.

7.2.7.1 If the request is for SVOA, use the SVOA Spiking Solution.

7.2.7.2 If the request is for SIM, use the SIM Spiking solution.

7.2.7.3 If the request is for SVOA Appendix IX, use the SVOA Appendix IX Spiking solution as well as the SVOA spiking solution

7.2.8 For each sample, rinse the original sample container with 60 mL of methylene chloride. Add this rinse to the separatory funnel.

7.2.9 Adjust the pH of the samples (including method blank, LCS/LCSD, and MS/MSD) to pH $\geq$ 11 with 10N NaOH after addition of surrogates and spikes. Stir with a glass stirring rod and check pH by tapping the glass stirring rod onto wide range pH paper. The pH must be $\geq$ 11. If the pH test strip does not clearly indicate the pH is greater than 11, narrow range pH paper must be used.

7.2.10 Add 60 mL of methylene chloride directly to the method blank and LCS/LCSD separatory funnels.

7.2.11 Extract the samples by shaking the funnel for two minutes, venting often, but gently, in a hood to release pressure. A mechanical shaker may be used, where samples are shaken for 3 minutes. Following each shake, allow phases to separate for at least 10 minutes. Drain the methylene chloride layer into an amber collection bottle.

7.2.12 If an emulsion forms, mechanical techniques must be employed to achieve maximum separation. Such means include swirling, centrifugation, and draining through a small separatory funnel. In certain instances, transferring the entire sample into a continuous liquid-liquid extractor (CLLE) may be the only alternative. If any such techniques are used, they must be noted in the extractions logbook, and the batch transferred to a CLLE batch with its own batch ID.

7.2.13 Add a second 60 mL aliquot of methylene chloride to the separatory funnel and extract for the second time (see 7.2.12 – 7.2.13). Collect the methylene chloride layer in the same amber collection bottle.

7.2.14 Repeat the extraction for a third time as described in 7.2.14.

7.2.15 Following the third shake, using a glass stirring rod, check the pH to ensure that it has remained at $\geq$ 11. If the pH has changed back to neutral range, it must be readjusted to $\geq$ 11 and the sample must be extracted at least one more time, adding the methylene chloride to the same amber bottle, that was previously used. If the pH has remained at a value $\geq$ 11, the pH is then adjusted to $\leq$ 2 with 1:1 H_2SO_4. Add enough 1:1 H_2SO_4 to adjust the pH to $\leq$ 2 with stirring. Use glass stirring rods to stir.

7.2.16 Add 60 mL methylene chloride and extract the samples three times in the same manner described in 7.2.11 – 7.2.13. Collect the methylene chloride layer in the same amber collection bottle used to collect the acid fraction.

7.2.17 Sample waste should be poured into the "n-lo" sattalite.

7.2.18 Proceed to Section 7.3 for extract concentration procedures.

7.3 CONCENTRATING THE EXTRACTS

For Methods 3510 and 3520, the combined fractions are concentrated to a final volume of 1.0 mL.

7.3.1 Rinse the K-D glassware (flask, concentration tube, and snyder column, including the ground glass joints on the flask and columns) three times with methylene chloride. Add two boiling chips to the K-D prior to final rinse. Also rinse the assembled funnels, filter paper, and granular sodium sulfate used for drying the extracts.

7.3.2 Transfer the methylene chloride extract to a K-D concentrator setup through a short stem funnel filled with 1-2 inches of sodium sulfate in fluted filter paper. This is the drying step, which is required to remove residual water from the extracts. Any large water layers must be removed by other means, prior to pouring through the sodium sulfate. After pouring all of the extract volume through the sodium sulfate, rinse the extract flask three times with ~ 2 – 3 mls of methylene chloride. Add the rinsings through the sodium sulfate to complete a quantitative transfer. Rinse the sodium sulfate with ~ 15 mls of methylene chloride and allow to drain

7.3.3 Transfer the label from the collection bottle or round bottom flask (for CLLE) to a K-D. Remove the funnel and attach a 3- or 4-ball macro Snyder column. Pre-wet the Snyder column with 1 mL of methylene chloride.

7.3.4 Place the K-D in a hot water bath (75-85°C). Gently swirl K-D in the water until boiling begins. At the proper distillation rate, the Snyder balls should chatter but the chambers should not flood with condensed solvent. The K-D should be kept in a vertical orientation while on the bath. When the apparent volume in the concentrator tube reaches ≈ 6 mL, remove the K-D from the water bath. Allow the K-D to cool for 10 minutes. Rinse the Snyder column lower joint with ≈ 1 mL of methylene chloride. Remove the Snyder column. Wipe off any water from the neck above the lower joint of the flask. Separate the K-D flask from the concentrator tube, rinsing the ground glass joint with ≈ 1 mL methylene chloride.

7.3.5 Reduce the methylene chloride extract in the concentrator tube to approximately 1 mL using the nitrogen blow-down apparatus. The bath temperature must be no higher than the boiling point of the solvent (39°C for

methylene chloride). Turn the gas to 3 psi. Be careful not to splash the extract out of the tube. During concentration on the N-evap, the internal wall of the concentrator tube and the N-evap sparging pipet must be rinsed down at least once or twice with ≈1 mL of methylene chloride. The solvent level in the concentrator tube must be positioned below the level of the water bath as much as possible to prevent water from condensing into the sample extract. As the extract volume is reduced, lower the N_2 sparging pipet closer to the surface of the extract to expedite the concentration. Note any problems or extract losses, if they occur, in the extractions logbook.

7.3.6 Reduce each extract to slightly less than 1 mL and then, using a 5 ¾" pasteur pipet, transfer the final extract and label to a 1.8 mL vial with PTFE-lined cap.

7.3.7 If at any time during the concentration process the concentrator tube goes dry, reextraction must occur immediately.

7.3.8 Using methylene chloride for a quantitative transfer, adjust the final volume of each extract to 1 mL. Use the 1 mL oil-filled reference vial for volume comparison.

7.3.9 Store in refrigerator until GC/MS analysis.

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

A method blank must be extracted for each and every item listed below:

- Each sample matrix (soil, water)
- Each day of extraction (24 hours midnight - midnight)
- Each extraction method or level
- Every 20 samples extracted in a 24-hour period

A laboratory control sample (LCS) is required for each and every item listed below:

- Each sample matrix
- Each extraction method or level
- Every extraction batch of twenty or fewer samples

Refer to the current revision of the applicable Katahdin SOP for analysis of semivolatiles for quality control acceptance criteria.

9.0 METHOD PERFORMANCE

Refer to the applicable analytical SOP.

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10.0 APPLICABLE DOCUMENTS/REFERENCES

Test Methods for Evaluating Solid Waste-Physical/Chemical Methods, Methods 3510 and 3520 (current revisions), SW-846 Third Edition, Updates I, II, IIA, and IIB, Revised January 1995, US EPA.

LIST OF TABLES AND FIGURES

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TABLE 1

SUMMARY OF METHOD MODIFICATIONS (METHOD 3510, current revision)

TOPIC	KATAHDIN SOP CA-502-06	METHOD 3510, current revision
Apparatus/Materials	1) 250 mL amber bottle or flask 2) 1.0 mL syringe 3) short stem funnels	1) 250 mL Erlenmeyer flask 2) 5.0 mL syringe 3) drying columns
Reagents		
Sample preservation/ handling		
Procedures	1) extract collection in amber bottle or Erlenmeyer flask 2) Add surrogate/spike to sample in CLLE 3) Extract for 3 minutes on mechanical shaker 4) extract three times at pH $\geq$ 11, then extract three times at pH $\leq$ 2. 5) extract dried using Na_2SO_4 in short stem funnels 6) Rinse the extract flask three times with ~ 2 – 3 mLs of methylene chloride then rinse the sodium sulfate with ~ 15 mLs of methylene chloride to complete a quantitative transfer 7) water bath temp 75-85 deg C 8) no apparatus height specification for concentration on water bath 9) sample removed from water bath when volume reaches ~6 mL 10) N bath temp no higher than 39 deg C	1) extract collection in Erlenmeyer flask 2) Add surrogate/spike directly to sample bottle 3) Extract by shaking vigorously for 1 - 2 minutes with periodic venting 4) extract three times at pH $\leq$ 2, then extract three times at pH $\geq$ 11. 5) extract dried using Na_2SO_4 in drying columns 6) Rinse the Erlenmeyer flask, which contained the solvent extract, with 20 - 30 mL of methylene chloride to complete the quantitative transfer 7) water bath temp 15-20 deg C above solvent boiling temp 8) partially immerse concentrator tube in water and lower apparatus to complete concentration in 10-20 min 9) sample removed from water bath when volume reaches 1 mL 10) N bath temp 35 deg C
QC - Spikes	1) Acid surrogate and spike components at 100 ug/mL; base/neutral surrogate and spike components at 50 ug/mL	1) Acid surrogate and spike components at 200 ug/mL; base/neutral surrogate and spike components at 100 ug/mL
QC - LCS	1) Acid surrogate and spike components at 100 ug/mL; base/neutral surrogate and spike components at 50 ug/mL	1) Acid surrogate and spike components at 200 ug/mL; base/neutral surrogate and spike components at 100 ug/mL

TITLE: PREPARATION OF AQUEOUS SAMPLES FOR EXTRACTABLE SEMIVOLATILE ANALYSIS

TABLE 1, continued

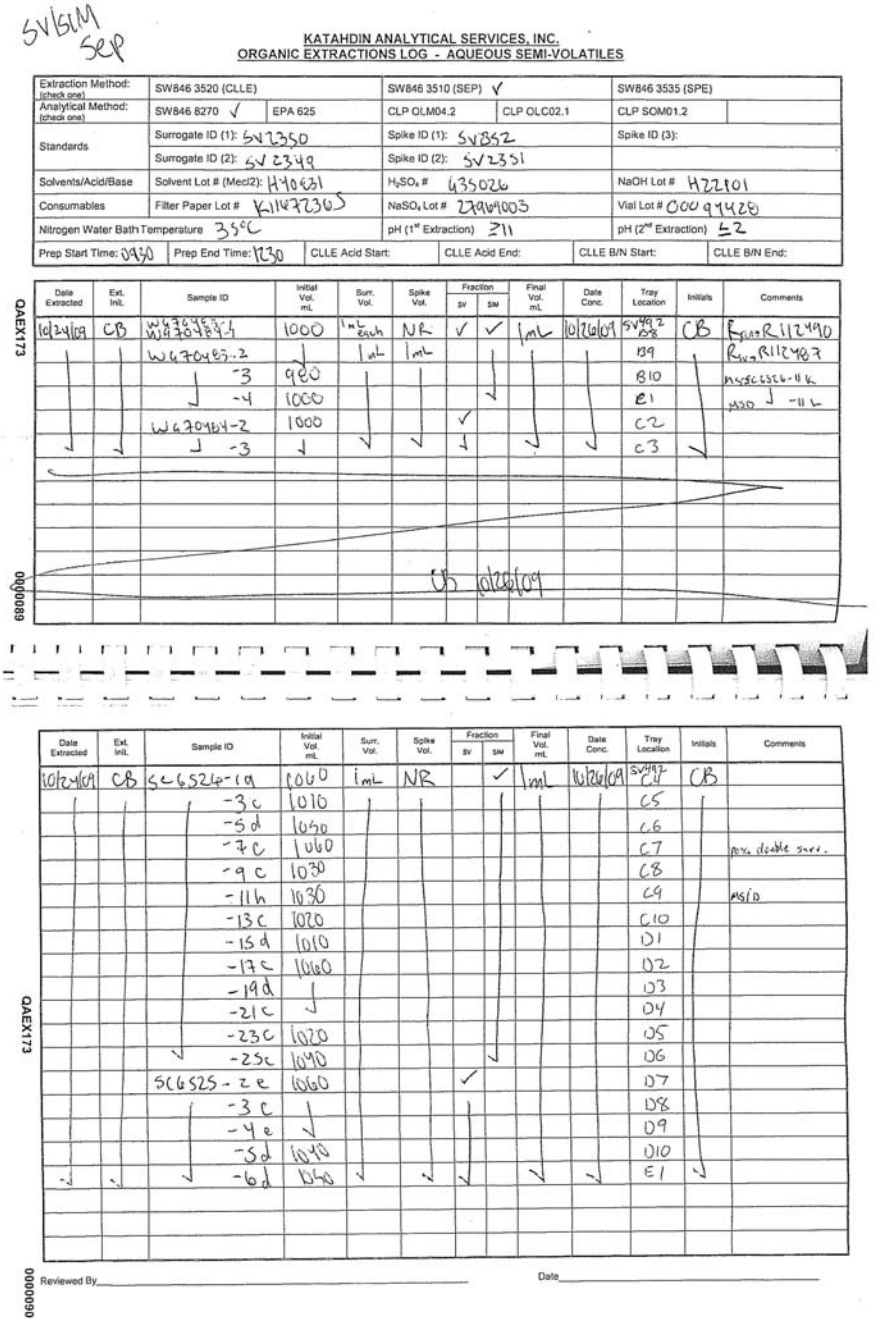
SUMMARY OF METHOD MODIFICATIONS (METHOD 3520, current revision)

TOPIC	KATAHDIN SOP CA-502-06	METHOD 3520, current revision
Apparatus/Materials	1) short stem funnels	1) drying columns
Reagents		
Sample preservation/ handling		
Procedures	1) Add surrogate/spike to sample in CLLE 2) Add approximately 500 - 600 mL of methylene chloride to the CLLE body 3) CLLE for 22 $\pm$ 2 hours 4) Extract dried using Na_2SO_4 in short stem funnels 5) Rinse the extract flask three times with ~ 2 – 3 mLs of methylene chloride then rinse the sodium sulfate with ~ 15 mLs of methylene chloride to complete a quantitative transfer 6) water bath temp 75-85 deg C 7) no apparatus height specification for concentration on water bath 8) sample removed from water bath when volume reaches ~6 mL 9) N bath temp no higher than 39 deg C	1) Add surrogate/spike directly to sample bottle 2) Add 300 - 500 mL of methylene chloride to the distilling flask of the extractor 3) CLLE for 18 - 24 hours 4) Extract dried using Na_2SO_4 in drying columns 5) Rinse the Erlenmeyer flask, which contained the solvent extract, with 20 - 30 mL of methylene chloride to complete the quantitative transfer 6) water bath temp 15-20 deg C above solvent boiling temp 7) partially immerse concentrator tube in water and lower apparatus to complete concentration in 10-20 min 8) sample removed from water bath when volume reaches 1 mL 9) N bath temp 35 deg C
QC - Spikes	1) Acid surrogate and spike components at 100 ug/mL; base/neutral surrogate and spike components at 50 ug/mL	1) Acid surrogate and spike components at 200 ug/mL; base/neutral surrogate and spike components at 100 ug/mL
QC - LCS	1) Acid surrogate and spike components at 100 ug/mL; base/neutral surrogate and spike components at 50 ug/mL	1) Acid surrogate and spike components at 200 ug/mL; base/neutral surrogate and spike components at 100 ug/mL

TITLE: PREPARATION OF AQUEOUS SAMPLES FOR EXTRACTABLE SEMIVOLATILE ANALYSIS

FIGURE 1

EXAMPLE OF SEMIVOLATILES LOGBOOK PAGE



SV/SIM Sep

KATAHDIN ANALYTICAL SERVICES, INC.
ORGANIC EXTRACTIONS LOG - AQUEOUS SEMI-VOLATILES

Extraction Method: (check one)	SW846 3520 (CLLE)		SW846 3510 (SEP) ✓		SW846 3535 (SPE)	
Analytical Method: (check one)	SW846 8270 ✓	EPA 625	CLP OLM04.2	CLP OLC02.1	CLP SOM01.2	
Standards	Surrogate ID (1): SV2350		Spike ID (1): SV852		Spike ID (3):	
	Surrogate ID (2): SV2349		Spike ID (2): SV2351			
Solvents/Acid/Base	Solvent Lot # (MeCl2): H40631		H_2SO_4 # 435026		NaOH Lot # H22101	
Consumables	Filter Paper Lot # K11472305		$NaSO_4$ Lot # 279069003		Vial Lot # 000 94428	
Nitrogen Water Bath Temperature 35°C			pH (1st Extraction) ≥11		pH (2nd Extraction) ≤2	
Prep Start Time: 0930	Prep End Time: 1230	CLLE Acid Start:	CLLE Acid End:	CLLE B/N Start:	CLLE B/N End:	

Date Extracted	Ext. Init.	Sample ID	Initial Vol. mL	Surr. Vol.	Spike Vol.	Fraction SV	Fraction SM	Final Vol. mL	Date Conc.	Tray Location	Initials	Comments
10/24/09	CB	WG470483-1	1000	1mL each	NR	✓	✓	1mL	10/26/09	SV492 C8	CB	Run=R112490
		WG470483-2	↓	1mL	1mL					B9		Run=R112487
		-3	980							B10		MS/SC6526-11h
		-4	1000				✓			B1		MSD ↓ -11h
		WG470484-2	1000			✓				C2		
		-3	↓	↓	↓	↓		↓	↓	C3	↓	

CB 10/26/09

Date Extracted	Ext. Init.	Sample ID	Initial Vol. mL	Surr. Vol.	Spike Vol.	Fraction SV	Fraction SM	Final Vol. mL	Date Conc.	Tray Location	Initials	Comments
10/24/09	CB	SC6526-1a	1060	1mL	NR		✓	1mL	10/26/09	SV492 C4	CB	
		-3c	1010							C5		
		-5d	1030							C6		
		-7c	1060							C7		90% dicable surr.
		-9c	1030							C8		
		-11h	1030							C9		MS/D
		-13c	1020							C10		
		-15d	1010							D1		
		-17c	1060							D2		
		-19d	↓							D3		
		-21c	↓							D4		
		-23c	1020							D5		
↓		-25c	1040				↓			D6		
		SC6525-2e	1060			✓				D7		
		-3c	↓			↓				D8		
		-4e	↓							D9		
		-5d	1040							D10		
↓	↓	-6d	1040	↓	↓	↓		↓	↓	E1	↓	

Reviewed By_____ Date_____

QAEX173 00000089

QAEX173 00000090

TITLE: PREPARATION OF AQUEOUS SAMPLES FOR EXTRACTABLE SEMIVOLATILE ANALYSIS

FIGURE 2

SOLVENT/REAGENT LOT CHECK LOGBOOK

SOLVENT:

LOT#:

DATE RECEIVED:

DATE CONCENTRATED:

CONCENTRATED BY:

PREP METHOD:

TRAY LOCATION:

ANALYZED BY:

PASS/FAIL:

TITLE: PREPARATION OF AQUEOUS SAMPLES FOR EXTRACTABLE SEMIVOLATILE ANALYSIS

FIGURE 3

LCS/MATRIX SPIKE COMPONENT LIST

BASE/NEUTRALS	
1-Methylnaphthalene	Bis (2-chloroethoxy) methane
1,1-Biphenyl	Bis (2-chloroethyl) ether
1,2,4-Trichlorobenzene	Bis (2-Chloroisopropyl) ether)
1,2-Dichlorobenzene	Bis(2-Ethylhexyl)adipate
1,3-Dichlorobenzene	Bis (2-ethylhexyl) phthalate
1,4-Dichlorobenzene	Butylbenzyl phthalate
1,4-Dioxane	Caprolactam
2,4-Dinitrotoluene	Carbazole
2,6-Dinitrotoluene	Chrysene
2-Chloronaphthalene	Dibenz (a, h) anthracene
2-Methylnaphthalene	Dibenzofuran
2-Nitroaniline	Diethyl phthalate
3,3'-Dichlorobenzidine	Diethyl adipate
3-Nitroaniline	Dimethyl phthalate
4-Bromophenylphenyl ether	Di-n-butylphthalate
4-Chloroaniline	Di-n-octyl phthalate
4-Chlorophenylphenyl ether	Fluoranthene
4-Nitroaniline	Fluorene
Acenaphthene	Hexachlorobenzene
Acenaphthylene	Hexachlorobutadiene
Acetophenone	Hexachlorocyclopentadiene
Aniline	Hexachloroethane
Anthracene	Indeno (1,2,3-cd) pyrene
Atrazine	Isophorone
Azobenzene	Naphthalene
Benzaldehyde	Nitrobenzene
Benzidine	N-Nitrosodimethylamine
Benzo (a) Anthracene	N-Nitroso-di-n-propylamine
Benzo (a) pyrene	N-Nitrosodiphenylamine
Benzo (b) fluoranthene	Phenanthrene
Benzo (ghi) perylene	p-toluidine
Benzo (k) fluoranthene	Pyrene
Benzyl alcohol	Pyridine

ACIDS		
2, 3, 4, 6-Tetrachlorophenol	2-Chlorophenol	Benzoic acid
2,4,5-Trichlorophenol	2-Methylphenol	Ethyl methanesulfonate
2,4,6-Trichlorophenol	2-Nitrophenol	Methyl methanesulfonate
2,4-Dichlorophenol	4,6-Dinitro-2-methylphenol	Pentachlorophenol
2,4-Dimethylphenol	4-Chloro-3-methylphenol	Phenol
2,4-Dinitrophenol	4-Methylphenol	
2,6-Dichlorophenol	4-Nitrophenol	

TITLE: PREPARATION OF AQUEOUS SAMPLES FOR EXTRACTABLE SEMIVOLATILE ANALYSIS

FIGURE 4

APPENDIX IX LCS/MATRIX SPIKE COMPONENT LIST

1,2,4,5-Tetrachlorobenzene	Hexachloropropene
1,3,5-Trinitrobenzene	Isodrin
1,4-Naphthoquinone	Isosafrole
1-Chloronaphthalene	Kepone
1-Naphthylamine	m-Dinitrobenzene
2,4-D	Methapyrilene
2-Acetyl aminofluorene	Methyl parathion
2-Naphthylamine	n-Nitrosodiethylamine
2-Picoline	n-Nitrosodi-n-butylamine
3,3-Dimethylbenzidine	n-Nitrosomethylethylamine
3-Methylcholanthrene	n-Nitrosomorpholine
4-Aminobiphenyl	n-Nitrosopyrrolidine
4-Nitroquinoline-1-oxide	n-Nitrotrosopiperidine
5-Nitro-o-toluidine	O,O,O-Triethyl phosphorothioate
7,12-Dimethylbenz(a)anthracene	o-Toluidine
a,a-Dimethylphenethylamine	Parathion
Acetophenone	p-Dimethylaminoazobenzene
Aramite	Pentachlorobenzene
Chlorobenzilate	Pentachloronitriobenzene
Diallate	Phenacetin
Dibenz(a,j)acridine	Phorate
Dimethoate	p-Phenylenediamine
Dinoseb	Pronamide
Diphenylamine	Safrole
Disulfoton	Silvex (2,4,5-TP)
Famphur	Sulfotep
Hexachlorophene	Thionazin

TITLE: PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT EXTRACTABLE SEMI-VOLATILES ANALYSIS

Prepared By: Mike Thomas Date: 09/96

Approved By:

Group Supervisor: Michael F. Thomas Date: 11/15/00

Operations Manager: JC Bruta Date: 10/25/00

QA Officer: Deborah J. Nadeau Date: 10.24.00

General Manager: Dennis P. Hogan Date: 11/16/00

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
01	Minor changes throughout. Clarifications to procedure section.	DN	10.24.00	10/24/00
02	Addition of compounds to Figure 2.	DN	3.28.02	3.28.02
03	Definitions added to section 1.1. Wording was added or changed to clarify sections 4, 5, 6, 7, 8 + 9. Minor changes throughout. New figures.	MRC	11.08.04	11.08.04
04	Updated Sect. 5.0 with current spike solutions prep. Removed section on medium level soil extraction. Replaced figure 3 and 4 with current LCS/MS spike components. Minor corrections to sect. 1.3, 4.24, 6.0 and 7.12. Updated logbook	LAD	04/06	04/06
05	Many changes made throughout, including but not limited to, waste information, updated spikes and surrogates, added SIM LCS/D and MS/D information, updated Table 1. Please refer to the QAM/SOP change form filed w/ SOP in QA for a detailed list of changes.	LAD	09/07	09/07

TITLE: PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT EXTRACTABLE SEMI-VOLATILES ANALYSIS

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
06	Updated logbook example. Added adipate compounds to fig. 2. Added necessity of recording lot numbers of consumables in logbook. Added to record the temperature of the nitrogen evaporation water bath.	LAD	07/08	07/08
07	Added requirement to add spike before Na_2SO_4. Changed N_2 water bath temperature from < 39°C to < 30°C. Removed respirator reference. Added KAEHS manual. Added KAS SOP CA-1008 reference for additional subsampling information.	LAD	02/09	02/09

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT EXTRACTABLE SEMI-VOLATILES ANALYSIS**

Please acknowledge receipt of this standard operating procedure by signing and dating both of the spaces provided. Return the bottom half of this sheet to the QA Department.

I acknowledge receipt of copy ___ of document **SOP CA-512-07**, titled **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT EXTRACTABLE SEMI-VOLATILES ANALYSIS.**

Recipient: _____ Date: _____

KATAHDIN ANALYTICAL SERVICES, INC.
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy ___ of document **SOP CA-512-07**, titled **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT EXTRACTABLE SEMI-VOLATILES ANALYSIS.**

Recipient: _____ Date: _____

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT EXTRACTABLE SEMI-VOLATILES ANALYSIS**

1.0 SCOPE AND APPLICATION

The purpose of this SOP is to describe the procedures and requirements for the preparation of solid samples for analysis of extractable semivolatile organic compounds. This SOP is specifically applicable to EPA Method 3550B in accordance with SW-846 Method 8270, current revision.

1.1 Definitions

METHOD BLANK (laboratory reagent blank): An artificial sample designed to determine if method analytes or other interferences are present in the laboratory environment, the reagents, or the apparatus. For aqueous samples, reagent water is used as a blank matrix; for soil samples, baked organic-free sand is used as a blank matrix. The blank is taken through the appropriate steps of the process.

LABORATORY CONTROL SAMPLE (LCS): A blank that has been spiked with the analyte(s) from an independent source, and is analyzed exactly like a sample. Its purpose is to determine whether the methodology is in control, and whether the laboratory is capable of making accurate and precise measurements. The matrix used should be phase matched with the samples and well characterized.

MATRIX SPIKE/MATRIX SPIKE DUPLICATE (MS/MSD): Predetermined quantities of stock solutions of certain analytes are added to a sample matrix prior to sample extraction and analysis. Samples are split into duplicates, spiked and analyzed. Percent recoveries are calculated for each of the analytes detected. The relative percent difference between the samples is calculated and used to assess analytical precision.

SURROGATES: Organic compounds which are similar to analytes of interest in chemical composition, extraction and chromatography, but which are not normally found in environmental samples. These compounds are spiked into all blanks, standards, samples and spiked samples prior to analysis. Percent recoveries are calculated for each surrogate.

1.2 Responsibilities

This method is restricted to use by, or under the supervision of analysts experienced in the extraction of samples for semivolatile analysis. Each analyst must demonstrate and document their ability to generate acceptable results with this method. Refer to Katahdin SOP QA-805, current revision, “Personnel Training”.

It is the responsibility of all Katahdin technical personnel involved i n the extraction of samples for semivolatile analysis to read and understand this SOP, adhere to the procedures outlined, and to properly document their d ata in the appropriate lab

notebook. Any deviations from the test or irregularities with the samples should also be recorded in the lab notebook and reported to the Department Manager or designated qualified data reviewer responsible for this data.

It is the responsibility of the Department Manager to oversee that members of their group follow this SOP, to ensure that their work is properly documented and to indicate periodic review of the associated logbooks.

1.3 Safety

Users of this procedure must be cognizant of inherent laboratory hazards, proper disposal procedures for contaminated materials and appropriate segregation of hazardous wastes. The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical should be treated as a potential health hazard. A reference file of material safety data sheets is available to all personnel involved in the chemical analys is. Everyone involved with the procedure must be familiar with the MSDSs for all the materials used in this procedure.

Each qualified analyst or technician must be familiar with Katahdin Analytical Environmental Health and Safety Manual including the Katahdin Hazardous Waste Plan and must follow appropriate procedures. These include the use of appropriate personal protective equipment (PPE) such as safety glasses, gloves and lab coats when working with chemicals or near an instrument and not taking food or drink into the laboratory. Each analyst should know the location of all safety equipment. Each analyst shall receive a safety orientation from their Department Manager, or designee, appropriate for the job functions they will perform.

1.4 Waste Disposal

Wastes generated during the preparation of samples must be disposed of in accordance with the procedures described in the current revision of the Katahdin Analytical Environmental Health and Safety Manual and SOP SD-903, “Sample Disposal,” current revision. Expired standards are lab packed, placed in the Katahdin hazardous waste storage area, and disposed of in accordance with this SOP.

Any methylene chloride solvent waste generated during the rinsing of glassware etc. should be disposed of in the “D” waste stream satellite accumulation area nearest the point of generation. Acetone and methanol are considered flammable waste, and should be disposed of in the “O” waste stream satellite accumulation area nearest the point of generation. Post-extraction soil samples and sodium sulfate waste should be disposed of in the soil with organics “I” waste stream satellite accumulation area nearest the point of generation. Please refer to the current revision of SOP CA-107 for the location of satellite waste accumulation areas.

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT EXTRACTABLE SEMI-VOLATILES ANALYSIS**

2.0 SUMMARY OF METHOD

A 30 gram portion of sediment/soil is mix ed with anhydrous powdered sodium sulfate and extracted with 1:1 methylene chloride/acetone (v/v) using an ultrasonic probe. The methylene chloride extract is dried and concentrated to a volume of 1.0 mL.

3.0 INTERFERENCES

Contaminants in solvents, reagents, glassware, and other sample processing hardware may cause method interferences such as discrete artifacts and/or elevated baselines in the total ion current profiles (TICPs). All of these materials routinely must be demonstrated to be free from interferences under the conditions of the analysis by running laboratory method blanks. Interferences caused by phthalate esters can pose a major problem in semivolatile organics analysis because many phthalates are also target analytes. Common flexible plastics contain varying amounts of phthalates that are easily extracted during laboratory operations, so cross-contamination of glassware frequently occurs when plastics are handled. Interferences from phthalates can best be minimized by avoiding the use of such plastics in the laboratory.

At no time may gloves that have not been tested for phthalates or gloves known to contain phthalates be used or stored in the organic extraction lab. Additionally, whenever possible plastic items in this lab must be replaced with metal or Teflon or other non-phthalate plastic substitute.

Special care should be taken to ensure clean glassware and apparatus are used, pre-rinsed with the appropriate solvent prior to use. Solvents should be analyzed prior to use to demonstrate that each lot is free of contaminants that may interfere with the analysis. Interferences coextracted from the samples will vary considerably from source to source. If analysis of an extracted sample is prevented due to inteferences, further cleanup of the sample extract may be needed to minimize interferences.

4.0 APPARATUS AND MATERIALS

Prior to use, all glassware must be rinsed three times with methylene chloride. Brand names and catalog numbers are included below for illustration purposes only.

4.1 Syringe - gas tight, 1.0 mL, solvent rinsed between each use.

4.2 Sonicator ultrasonic processor X L – Misonix (or equivalent) equipped with dual titanium 3/4" horn extenders for extracting two samples at a time.

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT EXTRACTABLE SEMI-VOLATILES ANALYSIS**

4.3 Powder funnels, 100 mm diameter, 35 mm stem

4.4 Kuderna-Danish (KD) apparatus - Concentrator tube - 10 mL
Evaporative flask - 500 mL
Snyder column - 3-ball macro

4.5 Filter paper, 7.0 cm, Whatman #4

4.6 Vacuum filtration flask - 500 mL Erlenmeyer

4.7 Buchner funnel, porcelain, Coors® with 85 mm plate diameter (or equivalent)

4.8 Beakers - 400 mL

4.9 Boiling chips - approximately 12 mesh, silicon carbide (carborundum or equivalent). Soxhlet extract overnight in methylene chloride.

4.10 Water bath - eight position concentric ring bath, or equivalent, equipped with a calibrated thermometer. The bath should be used in a hood.

4.11 Balance - capable of accurately weighing ± 0.1 g.

4.12 Vials and caps – 1.8 mL with PTFE/silicone septa and 12 mL with Teflon-lined caps for extracts designated for GPC cleanup.

4.13 Filter paper, 18.5 cm, Fisherbrand or Whatman 114V (or equivalent)

4.14 Pasteur pipets - disposable, 5 ¾ ".

4.15 Nitrogen evaporation apparatus.

4.16 Muffle oven – capable of maintaining 400 °C for baking glass wool and organic-free sand.

5.0 REAGENTS

5.1 Sodium Sulfate - anhydrous powdered and granular crystals, reagent grade, certified by the manufacturer/vendor as purified heating to 400°C prior to receipt by the laboratory. Solvent rinse immediately prior to use by rinsing three times with pesticide grade methylene chloride. (Jost Chemical anhydrous powder, catalog #2797 or equivalent, and Jost Chemical granular crystals, catalog #2796 or equivalent).

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT EXTRACTABLE SEMI-VOLATILES ANALYSIS**

5.2 Methylene chloride, methanol, and acetone - pesticide residue analysis grade or equivalent. Methylene chloride and acetone are evaluated by lot prior to use by concentration of approximately 400 mL to 1.0 mL followed by GC/MS analysis. The lot numbers of all solvents used during an extraction must be recorded in the extraction logbook.

5.3 Organic-free sand, purified by baking at 400 °C. Method blanks serve as checks on the baked sand.

5.4 Base/Neutral and Acid (SVOA) Surrogate Spiking Solution - Surrogate standards are added to all samples and calibration solutions. Prepare a surrogate standard spiking solution that contains the following compounds at the indicated concentrations in acetone.

Compound	Conc.
phenol-$_{d6}$	100 ug/mL
2,4,6-tribromophenol	100 ug/mL
2-fluorophenol	100 ug/mL
nitrobenzene-$_{d5}$	50 ug/mL
p-terphenyl-$_{d14}$	50 ug/mL
2-fluorobiphenyl	50 ug/mL

Store the spiking solution at -10°C to -20°C in Teflon-sealed containers. These solutions must be replaced after six months, or sooner if comparison with quality control check samples indicates a problem. If reanalysis of a method blank still indicates surrogates out of criteria, a new surrogate solution must be used.

5.5 SIM Surrogate Spiking Solution- Surrogate Standards are added to all samples and calibration solutions. Prepare a surrogate solution that contains the following compounds at a concentration of 2 ug/mL in acetone.

Compound	Conc. ug/mL
Fluorene-d10	2.0 ug/mL
2-Methylnaphthalene-d10	2.0 ug/mL
Pyrene-d10.	2.0 ug/mL
2,4-Dibromophenol	2.0 ug/mL

Store the spiking solution at -10°C to -20°C in Teflon-sealed containers. These solutions must be replaced after six months, or sooner if comparison with quality control check samples indicates a problem. If reanalysis of a method blank still indicates surrogates out of criteria, a new surrogate solution must be used.

5.6 Base/Neutral and Acid (SVOA)_ Matrix Spike/Lab Control Sample Spiking Solution - Prepare a spiking solution in methanol that contains the compounds listed in Figure 2 at a concentration of 50 ug/mL for base/neutrals and 100 ug/mL for acids. Store the

spiking solution at -10°C to -20°C in Teflon-sealed containers. These solutions must be replaced after six months, or sooner if comparison with quality control check samples indicates a problem.

5.7 Base/Neutral and Acid (SVOA APPENDIX IX) Matrix Spike/Lab Control Sample Spiking Solution. Prepare a spiking solution in methanol that contains the compounds listed in Figure 3 at a concentration of 100 µg/mL for each compound. Store the spiking solution at -10°C to -20°C in Teflon-sealed containers. These solutions must be replaced after six months, or sooner if comparison with quality control check samples indicates a problem

5.8 Base/Neutral (SIM) Matrix Spike/ Lab Control Sample Spike Solution for SIM-SVOA. Prepare a spiking solution in methanol that contains the compounds listed in Figure 2 at a concentration of 2 ug/mL for base/neutral. Take out 1.0 mL of Base/Neutral and Acid Matrix Spike/Lab Control Spiking Solution for SVOA and dilute it to 25.0 mL in methanol. Store the solution Spiking at -10°C to -20°C in Teflon-sealed containers. These solutions must be replaced after six months, or sooner if comparison with quality control check samples indicates a problem.

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

Sediment/soil samples must be collected in a soil jar and must be maintained at 4°C (±2°C).

Holding time for extraction of sediment/soil samples for Method 3550 is 14 days from date of sample collection, although the analyst should be aware that actual holding times employed may be project/program specific.

Store all extracts at 4°C (±2°C) in the dark in labeled Teflon-sealed containers. See SOP SD-902, "Sample Receipt and Internal Control," current revision, for storage areas and temperature maintenance procedures.

7.0 PROCEDURES

Some solid samples may need to be cleaned up to reduce matrix interferences. The cleanup procedure employed is gel permeati on chromatography (GPC). The organic department manager should be consulted to determine if a particular sample should be subjected to further cleanup procedures; the decision should consider sample history, sample appearance, and project/client needs.

Sign chain-of-custody when removing and replacing samples in storage locations, and fill out the sample preparation/extraction log with the necessary information before starting the extraction. Prerinse all glassware three times with methylene chloride.

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT EXTRACTABLE SEMI-VOLATILES ANALYSIS**

7.1 Decant and discard any water layer on a sediment sample. Mix with a stainless steel spatula to ensure homogeneity of the sample. If the sample container is full to the extent that stirring the sample is impractical, try to remove the "best representative" aliquot from the jar based on color, particle size, moisture, etc. Remove any foreign objects such as sticks, leaves, and rocks, and note actions taken in the appropriate extraction logbook. Please refer to the current revision of Katahdin Analytical Services SOP CA-108, "Basic Laboratory Technique ", for more detailed guidance on subsampling to ensure reproducibility.

7.2 The following steps should be performed rapidly to avoid loss of the more volatile extractable. Weigh out a 30.0 ± 0.05 g portion of sample into a labeled 400-mL beaker. Record sample weight to the nearest 0.1 g in appropriate extraction logbook. Refer to Add between 30 g and 60 g of anhydrous powdered sodium sulfate as required for producing a "free-flowing" mixture. The amount of sodium sulfate added will depend upon the moisture content of the sample (e.g., low moisture content will require less sodium sulfate). Mix well with a spatula. Keep the spatula in the sample beaker and cover the beaker with aluminum foil.

7.3 A method blank must be prepared with each extraction batch, not to exceed 20 client samples. To prepare a method blank, weigh out one 30.0 ± 0.05 g portion of purified sand in a labeled 400 mL beaker. Refer to sections 7.7 and 7.7 for spike and surrogate addition instructions. Add 60 g sodium sulfate and mix well. Although a "free-flowing" mixture can be achieved with less than 60 g sodium sulfate, the method blank must contain 60 g in order to evaluate the sodium sulfate as a potential source of contamination.

7.4 A laboratory control sample (LCS) must be prepared with each extraction batch, not to exceed 20 client samples. To prepare LCS, weigh out one 30.0 ± 0.05 g portion of purified sand in a labeled 400 mL beaker. Refer to sections 7.7 and 7.7 for spike and surrogate addition instructions. Add 30 g sodium sulfate and mix well. If combined SIM-SVOA analysis is requested, a separate LCS must be prepared for each analysis. If an MS/MSD pair is not extracted on a particular day, an LCS/LCSD pair may be required in order to meet client-specific or program-specific requirements. This information will be disseminated from the project manager or Department Manager.

7.5 A matrix spike/matrix spike duplicate (MS/MSD) set must be prepared for every 20 samples. To prepare MS/MSD, weigh out 30.0 ± 0.05 g portions of the sample designated for MS/MSD into each of two labeled 400 mL beakers. Record sample weights to nearest 0.1 g in appropriate extraction logbook. Refer to sections 7.7 and 7.7 for spike and surrogate addition instructions. Add 30 - 60 g sodium sulfate to each to produce a free-flowing mixture, and mix well. If combined SIM-SVOA analysis is requested, a separate MS/MSD must be prepared for each analysis.

7.6 Record all weights to one decimal place in the extraction logbook.

7.7 To all samples, method blank, LCS/LCSD, and MS/MSD add 1.0 mL of the appropriate base/neutral and acid surrogate spiking solution listed below using the pre-rinsed 1.0 mL gas tight syringe. The surrogate spike should be added **prior** to the addition of the sodium sulfate. Record surrogate spike volume and identification code in extraction logbook. Thoroughly rinse syringe with solvent prior to using it for another spiking solution.

7.7.1 If the request is for SVOA or SVOA Appendix IX , use the SVOA surrogate solution (sect. 5.4).

7.7.2 If the request is for SIM, use the SIM surrogate solution (sect. 5.5).

7.7.3 If the request is for SIM-SVOA, use both the SIM and SVOA surrogate solutions. NOTE: Separate LCS/LCSD and/or MS/MSD are needed for each analysis and should be spiked with the appropriate surrogate.

7.8 To the LCS/LCSD and the MS/MSD add 1.0 mL of the appropriate base/neutral and acid (SVOA) matrix spike/LCS spiking solution listed below using a 1.0 mL gas tight syringe. The LCS/MS spike should be added **prior** to the addition of the sodium sulfate. Record the matrix spike/LCS spiking solution volume and identification code in extraction logbook. Thoroughly rinse the syringe with solvent when spiking is completed.

7.8.1 If the request is for SVOA, add 1 mL of SVOA Spiking Solution (sect 5.6).

7.8.2 If the request is for SIM, add 1 mL SIM Spiking solution (sect 5.8).

7.8.3 If the request is for SVOA and SIM, add 1mL of SVOA Spiking Solution and 1 mL SIM Spiking solution (sect 5.6 and 5.8).

7.8.4 If the request is for SVOA Appendix IX , add 1mL of SVOA Spiking Solution and 1 mL of SVOA Appendix IX Spiking solution (sect 5.6 and 5.7).

7.9 To assure optimum operation and maximum energy output, the sonicators must be tuned daily prior to extracting samples. The following tuning procedure must be performed with the sonicator probes vibrating in air.

7.9.1 Turn OUTPUT CONTROL knob counter-clockwise to zero. This automatically switches the duty cycle to continuous mode.

7.9.2 Press and hold down the power switch to on.

7.9.3 Press and hold down the TUNE switch. Check if the counter is less or equal to 20%; otherwise, rotate the Tuning Knob (tuning button) clockwise until a reading of 20%or less is obtained.

7.9.4 Release the TUNE switch.

7.9.5 Turn OUTPUT CONTROL KNOB counter-clockwise to 50 and the power switch off.

7.9.6 Confirm that the sonicators were tuned by recording the date and/or percent in the extractions logbook.

7.10 Prior to extracting any samples, ensure that the sonicator probes are decontaminated by rinsing three times with a methylene chloride wash bottle. Collect the waste in a waste beaker. It may sometimes be necessary to wipe the upper part of each probe with a methylene chloride dampened KimWipe. Repeat this decontamination step between each sample on each probe.

7.11 To the mixed and spiked blank and LCS, add 100 mL of the 1:1 methylene chloride/acetone (V/V) solution and proceed with steps 7.11 through 7.14. Record the lot numbers of the solvents in the extraction logbook.

7.12 It may be necessary at this time to stir the sample/sodium sulfate mixture with the spatula to loosen up the mixture prior to extracting. Rinse the spatula with methylene chloride and collect the rinsing into a correspondent beaker. Position the beaker in the ultrasonic cell disruptor so that the bottom surface of the tip of the 3/4 inch disruptor horn is about halfway below the surface of the solvent and above the sediment layer.

7.13 Sonicate for 3 minutes with the output control knob set at 10, and mode switch on "pulsed" and % duty cycle knob set at 50%. While the mixture is sonicating, one should be able to see all, or most of the material, moving in the beaker under the influence of the energized probes. If not, stir the mixture again.

7.14 Prepare a filter flask fitted with a Buchner funnel. The Buchner funnel should contain a 7.0 cm Whatman #4 filter. Prerinse the flask, funnel and filter with methylene chloride and discard rinsings into solvent waste container. Decant extract into the filter flask and Buchner funnel. A vacuum pump may be used to facilitate filtration or the extract may be gravity filtered. The lot number of the filter paper must be written ti the extraction logbook.

7.15 Repeat the extraction two more times (sec 7.11 – 7.14) using 100 mL portions of 1:1 methylene chloride: acetone. Before each extraction, make certain that the sodium sulfate is still free-flowing and not a consolidated mass. As required, break up large

lumps with the spatula. Decant the extraction solvent into the Buchner funnel after each sonication. On the final sonication, pour the entire sample contents into the Buchner funnel and rinse thoroughly with methylene chloride to complete the quantitative transfer of the extract. Use the vacuum pump to pull all the extract into the flask

CONCENTRATION OF LOW LEVEL EXTRACTS

7.16 Rinse the K-D glassware (flask, concentration tube, and Snyder column, including the ground glass joints on the flask and columns) three times with methylene chloride before assembling. Add two boiling chips to the K-D. Insert fluted 18.5 cm filter papers into short stem powder funnels and add ~ 2 inches of sodium sulfate crystals. Rinse the sodium sulfate in the assembled funnels with ~20-30 mLs of methylene chloride. Place the assembled K-D's under the funnels. The lot number of the filter paper must be written ti the extraction logbook.

7.17 Transfer the extracts to the K-D concentrator setups through the sodium sulfate in the funnels. This is the drying step, which is required to remove residual water from the extracts. Any large water layers must be removed by other means, prior to pouring through the sodium sulfate. After pouring all of the extract volume through the sodium sulfate, rinse the extract flask three times with ~ 2 – 3 mls of methylene chloride. Add the rinsings through the sodium sulfate to complete a quantitative transfer. Rinse the sodium sulfate with ~ 15 mls of methylene chloride and allow to drain.

7.18 If samples are to be GPC'd, refer to the current revision of Katahdin SOP CA-513, Extract Cleanup Using Gel Permeation Chromatography, for appropriate concentrating procedures.

7.19 If samples are not to be GPC'd, follow Steps 7.19 through 7.24 to concentrate extracts to final volume of 1 mL. Otherwise proceed to GPC cleanup procedure as described in the current revision of Katahdin SOP CA-513.

7.20 Transfer the labels from the extract flasks to the K-Ds. Remove the funnel and attach a 3-ball macro Snyder column. Pre-wet the Snyder column with 1 mL of methylene chloride.

7.21 Place the K-D in a hot water bath (75-85°C). Gently swirl K-D in the water until boiling begins. At the proper distillation rate, the Snyder balls should chatter but the chambers should not flood with condensed solvent. The K-D should be kept in a vertical orientation while on the bath. When the apparent volume in the concentrator tube reaches ≈ 6 mL, remove the K-D from the water bath. **Do not allow the evaporator to go dry. If the sample extract does go dry, re-extraction must occur immediately.** Allow the K-D to cool for 10 minutes. Rinse the Snyder column lower joint with ≈ 1 mL of methylene chloride. Remove the

Snyder column. Wipe off any water from the neck above the lower joint of the flask. Separate the K-D flask from the concentrator tube, rinsing the ground glass joint with ≈ 1 mL methylene chloride.

7.22 Reduce the extract in the concentrator tube to approximately 1 mL using the nitrogen blow-down apparatus. The bath temperature must be < 30°C. Turn the gas to 3 psi. Be careful not to splash the extract out of the tube. During concentration on the N-evap, the internal wall of the concentrator tube and the N-evap sparging pipet must be rinsed down at least once or twice with ≈1 mL of methylene chloride. The solvent level in the concentrator tube must be positioned below the level of the water bath as much as possible to prevent water from condensing into the sample extract. As the extract volume is reduced, lower the N_2 sparging pipet closer to the surface of the extract to expedite the concentration. Record the temperature of the water in the nitrogen evaporation water bath in the logbook also note any problems or extract losses, if they occur, in the extractions logbook.

7.23 When the apparent volume reaches slightly less than 1 mL, remove the concentrator tube and allow it to cool.

7.24 Complete the quantitative transfer of the extract to a 1.8 mL vial by using methylene chloride. Adjust the volume of the methylene chloride extract to 1.0 mL using the 1.8 mL reference vial for volume comparison.

7.25 Label the vial with lab sample number, extraction date, matrix and analysis. Store extract vials at a temperature of 4 ± 2 °C until ready for analysis. Indicate in the extraction logbook the box number and “tray location” of the individual extract vials.

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

A method blank must be extracted for each and every item listed below:

- Each sample matrix
- Each extraction method or level
- Every extraction batch of twenty or fewer samples

A laboratory control sample (LCS) is required for each and every item listed below:

- Each sample matrix
- Each extraction method or level
- Every extraction batch of twenty or fewer samples

Refer to the current revision of the applicable Katahdin SOP for analysis of Semivolatile Organics for quality control acceptance criteria.

Each extractions analyst must demonstrate proficiency in performing the extractions that prepare samples for analysis. Demonstration consists of preparation (extraction), by the analyst, of at least four aliquots which are then analyzed according to the analytical method in question. These QC samples must meet all quality control acceptance limits. Demonstration must be documented by use of a form which summarizes the results of the analysis of these aliquots, calculated percent recoveries, and standard deviation. Demonstration of proficiency must be done one time per analyst initially and then annually thereafter. Refer to SOPs QA-805 and QA-807, current revision.

If, upon analysis of the extracted samples, it is discovered that quality control acceptance criteria have not been met, all associated samples must be evaluated against all the QC. In some cases data may be reported, perhaps with narration, while in other cases, other corrective action may be taken. The corrective actions may include re-extraction of the samples associated with the quality control sample that did not meet acceptance criteria, or may include making new reagents and standards if the standardization is suspect. These decisions are based on holding time considerations, client and project specific Data Quality Objectives and on review of chromatograms. The supervisor, Operations Manager, and/or Quality Assurance Officer may be consulted to evaluate data. Some samples may not be able to be reanalyzed within hold time. In these cases "qualified" data with narration may be advisable after consultation with the client.

Much of the work performed at the lab is analyzed in accordance with specific QC requirements spelled out in a project specific Quality Assurance Project Plan (QAPP) or in a program specific Quality Systems Manual (QSM). The reporting limits, acceptance criteria and/or corrective actions may be different than those specified in this SOP. In these cases the appropriate information will be communicated to the Department Manager and/or senior chemists before initiation of the analyses so that specific product codes can be produced for the project. In addition, the work order notes for each project will describe the specific QAPP or QSM to be followed.

9.0 METHOD PERFORMANCE

The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero. The MDLs are determined annually per type of instrument and filed with the Organic Department Manager and with the QAO. Refer to the current revision of Katahdin SOP QA-806, Method Detection Limit and Instrument Detection Limit Studies, for procedures on determining the MDL.

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT EXTRACTABLE SEMI-VOLATILES ANALYSIS**

Refer to the applicable analytical SOP for other method performance parameters and requirements.

10.0 APPLICABLE DOCUMENTS/REFERENCES

Test Methods for Evaluating Solid Waste-Physical/Chemical Methods, Method 3550C, USEPA SW-846, Third Edition, Update IV, February 2007.

LIST OF TABLES AND FIGURES

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT EXTRACTABLE SEMI-VOLATILES ANALYSIS**

TABLE 1

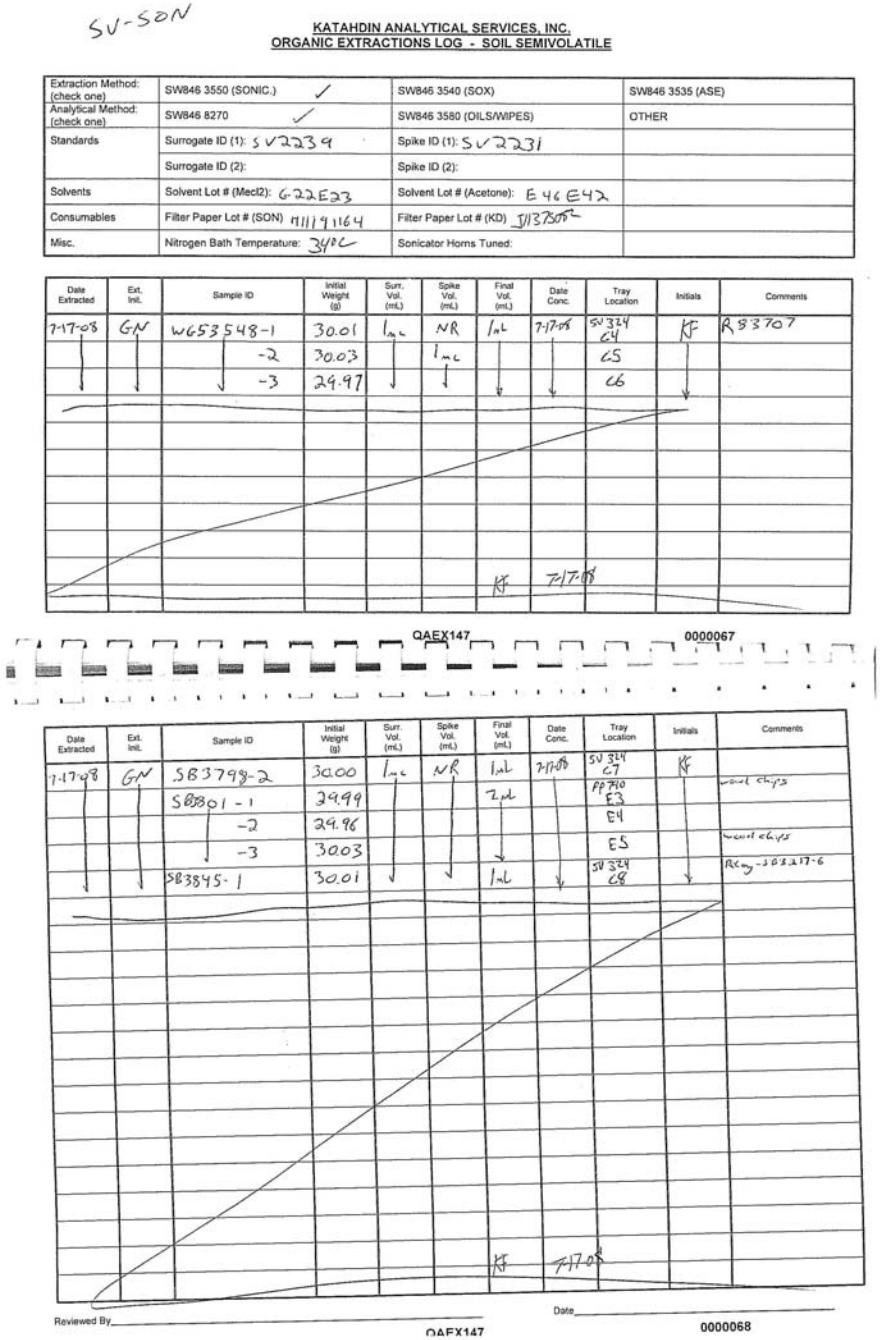
SUMMARY OF METHOD MODIFICATIONS

TOPIC	KATAHDIN SOP CA-512-07	METHOD 3550, current revision
Apparatus/Materials	1) short stem funnels	1) drying columns
Reagents		
Sample preservation/ handling		
Procedures	1) extract dried using Na_2SO_4 in short stem funnels 2) place sonicator horns ½ way between the surface of the solvent and the sediment layer 3) no apparatus height specification for concentration on water bath 4) water bath at 75-85 deg C 5) sample removed from water bath when volume reaches ~6 mL	1) extract dried using Na_2SO_4 in drying columns 2) place sonicator horns ½ inch below the solvent surface but above sediment layer 3) partially immerse concentrator tube in water and lower apparatus to complete concentration in 10-15min 4) water bath at 80-90 deg C 5) sample removed from water bath when volume reaches 1-2 mL
QC - Spikes	1) Acid surrogate and spike components at 100 ug/mL; base/neutral surrogate and spike components at 50 ug/mL	1) Acid surrogate and spike components at 200 ug/mL; base/neutral surrogate and spike components at 100 ug/mL
QC - LCS	1) Acid surrogate and spike components at 100 ug/mL; base/neutral surrogate and spike components at 50 ug/mL	1) Acid surrogate and spike components at 200 ug/mL; base/neutral surrogate and spike components at 100 ug/mL
QC - Accuracy/Precision		
QC - MDL		

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT EXTRACTABLE SEMI-VOLATILES ANALYSIS**

FIGURE 1

EXAMPLE OF LOGBOOK PAGE



SV-SON

KATAHDIN ANALYTICAL SERVICES, INC.
ORGANIC EXTRACTIONS LOG - SOIL SEMIVOLATILE

Extraction Method: (check one)	SW846 3550 (SONIC.) ✓	SW846 3540 (SOX)	SW846 3535 (ASE)
Analytical Method: (check one)	SW846 8270 ✓	SW846 3580 (OILS/WIPES)	OTHER
Standards	Surrogate ID (1): SV2239	Spike ID (1): SV2231	
	Surrogate ID (2):	Spike ID (2):	
Solvents	Solvent Lot # (MeCl2): G22E23	Solvent Lot # (Acetone): E46E42	
Consumables	Filter Paper Lot # (SON) 71119164	Filter Paper Lot # (KD) 7113805	
Misc.	Nitrogen Bath Temperature: 34°C	Sonicator Horns Tuned:	

Date Extracted	Ext. Init.	Sample ID	Initial Weight (g)	Surr. Vol. (mL)	Spike Vol. (mL)	Final Vol. (mL)	Date Conc.	Tray Location	Initials	Comments
7-17-08	GN	WG53548-1	30.01	1mL	NR	1mL	7-17-08	SV324 C4	KF	R83707
		-2	30.03		1mL			C5		
		-3	29.97					C6		
						KF	7/17/08			

QAEX147 0000067

Date Extracted	Ext. Init.	Sample ID	Initial Weight (g)	Surr. Vol. (mL)	Spike Vol. (mL)	Final Vol. (mL)	Date Conc.	Tray Location	Initials	Comments
7-17-08	GN	SB3798-2	30.00	1mL	NR	1mL	7-17-08	SV324 C7	KF	
		SB3801-1	29.99			2mL		PP710 E3		Used chips
		-2	29.96					E4		
		-3	30.03					E5		Used chips
		SB3845-1	30.01			1mL		SV324 C8		Reuse SB3217-6
						KF	7/17/08			

Reviewed By_____ Date_____

QAEX147 0000068

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT EXTRACTABLE SEMI-VOLATILES ANALYSIS**

FIGURE 2

LCS/MATRIX SPIKE COMPONENT LIST

BASE/NEUTRALS	
1-Methylnaphthalene	Bis (2-chloroethoxy) methane
1,1-Biphenyl	Bis (2-chloroethyl) ether
1,2,4-Trichlorobenzene	Bis (2-Chloroisopropyl) ether)
1,2-Dichlorobenzene	Bis (2-ethylhexyl) adipate
1,3-Dichlorobenzene	Bis (2-ethylhexyl) phthalate
1,4-Dichlorobenzene	Butylbenzyl phthalate
1,4-Dioxane	Caprolactam
2,4-Dinitrotoluene	Carbazole
2,6-Dinitrotoluene	Chrysene
2-Chloronaphthalene	Dibenz (a, h) anthracene
2-Methylnaphthalene	Dibenzofuran
2-Nitroaniline	Diethyl adipate
3,3'-Dichlorobenzidine	Diethyl phthalate
3-Nitroaniline	Dimethyl phthalate
4-Bromophenylphenyl ether	Di-n-butylphthalate
4-Chloroaniline	Di-n-octyl phthalate
4-Chlorophenylphenyl ether	Fluoranthene
4-Nitroaniline	Fluorene
Acenaphthene	Hexachlorobenzene
Acenaphthylene	Hexachlorobutadiene
Acetophenone	Hexachlorocyclopentadiene
Aniline	Hexachloroethane
Anthracene	Indeno (1,2,3-cd) pyrene
Atrazine	Isophorone
Azobenzene	Naphthalene
Benzaldehyde	Nitrobenzene
Benzidine	N-Nitrosodimethylamine
Benzo (a) Anthracene	N-Nitroso-di-n-propylamine
Benzo (a) pyrene	N-Nitrosodiphenylamine
Benzo (b) fluoranthene	Phenanthrene
Benzo (ghi) perylene	p-toluidine
Benzo (k) fluoranthene	Pyrene
Benzyl alcohol	Pyridine

ACIDS		
2, 3, 4, 6-Tetrachlorophenol	2-Chlorophenol	Benzoic acid
2,4,5-Trichlorophenol	2-Methylphenol	Ethyl methanesulfonate
2,4,6-Trichlorophenol	2-Nitrophenol	Methyl methanesulfonate
2,4-Dichlorophenol	4,6-Dinitro-2-methylphenol	Pentachlorophenol
2,4-Dimethylphenol	4-Chloro-3-methylphenol	Phenol
2,4-Dinitrophenol	4-Methylphenol	
2,6-Dichlorophenol	4-Nitrophenol	

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT EXTRACTABLE SEMI-VOLATILES ANALYSIS**

FIGURE 3

APPENDIX IX LCS/MATRIX SPIKE COMPONENT LIST

1,2,4,5-Tetrachlorobenzene	Hexachloropropene
1,3,5-Trinitrobenzene	Isodrin
1,4-Naphthoquinone	Isosafrole
1-Chloronaphthalene	Kepone
1-Naphthylamine	m-Dinitrobenzene
2,4-D	Methapyrilene
2-Acetyl aminofluorene	Methyl parathion
2-Naphthylamine	n-Nitrosodiethylamine
2-Picoline	n-Nitrosodi-n-butylamine
3,3-Dimethylbenzidine	n-Nitrosomethylethylamine
3-Methylcholanthrene	n-Nitrosomorpholine
4-Aminobiphenyl	n-Nitrosopyrrolidine
4-Nitroquinoline-1-oxide	n-Nitrotrosopiperidine
5-Nitro-o-toluidine	O,O,O-Triethyl phosphorothioate
7,12-Dimethylbenz(a)anthracene	o-Toluidine
a,a-Dimethylphenethylamine	Parathion
Acetophenone	p-Dimethylaminoazobenzene
Aramite	Pentachlorobenzene
Chlorobenzilate	Pentachloronitriobenzene
Diallate	Phenacetin
Dibenz(a,j)acridine	Phorate
Dimethoate	p-Phenylenediamine
Dinoseb	Pronamide
Diphenylamine	Safrole
Disulfoton	Silvex (2,4,5-TP)
Famphur	Sulfotep
Hexachlorophene	Thionazin

TITLE: PREPARATION OF AQUEOUS SAMPLES FOR PESTICIDES/PCBs ANALYSIS

Prepared By: Mike Thomas Date: 8/96

Approved By:

Group Supervisor: Michael F. Thomas Date: 11/15/00

Operations Manager: JCBurton Date: 10/25/00

QA Officer: Deborah J. Nadeau Date: 10·23·00

General Manager: Dennis F. Kuhn Date: 11/16/00

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
01	Miner changes throughout. clarifications to procedure section.	DN	10·23·00	
02	Addition of SPE Procedure. minor changes throughout Added wording to sections 6 and 8	LAD	013105	013105
03	Added separate QC for Pest. and PCB. updated concentration procedure to reflect current practices. Changes in wording for clarification. Update Logbook page	LAD	04/06	04/06
04	Added waste generated and disposal info. Added missing definitions. Updated SPE extraction procedure. Updated Table 1 and 2. Added Table 3.	LAD	09/07	09/07
05	Updated logbook example. Added logbook requirements	LAD	09/08	09/08

TITLE: PREPARATION OF AQUEOUS SAMPLES FOR PESTICIDES/PCBs ANALYSIS

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
06	Added information for determining initial volume. Added reference to CA-108. Added clarification for LCS/D and MS/D sets for PEST/PCB analysis. Minor changes to reflect current techniques	LAD	10/09	10/09

TITLE: PREPARATION OF AQUEOUS SAMPLES FOR PESTICIDES/PCBs ANALYSIS

Please acknowledge receipt of this standard operating procedure by signing and dating both of the spaces provided. Return the bottom half of this sheet to the QA Department.

I acknowledge receipt of copy _____ of document **SOP CA-515-06,** titled **PREPARATION OF AQUEOUS SAMPLES FOR PESTICIDES/PCBs ANALYSIS**.

Recipient: _____ Date: _____

KATAHDIN ANALYTICAL SERVICES, INC.
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy _____ of document **SOP CA-515-06,** titled **PREPARATION OF AQUEOUS SAMPLES FOR PESTICIDES/PCBs ANALYSIS**.

Recipient: _____ Date: _____

TITLE: PREPARATION OF AQUEOUS SAMPLES FOR PESTICIDES/PCBs ANALYSIS

1.0 SCOPE AND APPLICATION

The purpose of this SOP is to describe the procedures utilized by Katahdin Analytical Services, Inc. laboratory personnel for the preparation of aqueous samples prior to analysis for pesticides/PCBs by GC/ECD. It includes extraction of water samples by separatory funnel, continuous liquid-liquid, and solid phase extraction methods (EPA Methods 3510, 3520, 3535A, and EPA Method 608 current revisions).

1.1 Definitions

METHOD BLANK (laboratory reagent blank): An artificial sample designed to determine if method analytes or other interferences are present in the laboratory environment, the reagents, or the apparatus. For aqueous samples, reagent water is used as a blank matrix; for soil samples, baked organic-free sand is used as a blank matrix. The blank is taken through the appropriate steps of the process.

LABORATORY CONTROL SAMPLE (LCS): A blank that has been spiked with the analyte(s) from an independent source, and is analyzed exactly like a sample. Its purpose is to determine whether the methodology is in control, and whether the laboratory is capable of making accurate and precise measurements. The matrix used should be phase matched with the samples and well characterized.

MATRIX SPIKE/MATRIX SPIKE DUPLICATE (MS/MSD): Predetermined quantities of stock solutions of certain analytes are added to a sample matrix prior to sample extraction and analysis. Samples are split into duplicates, spiked and analyzed. Percent recoveries are calculated for each of the analytes detected. The relative percent difference between the samples is calculated and used to assess analytical precision.

SURROGATES: Organic compounds which are similar to analytes of interest in chemical composition, extraction and chromatography, but which are not normally found in environmental samples. These compounds are spiked into all blanks, standards, samples and spiked samples prior to analysis. Percent recoveries are calculated for each surrogate.

1.2 Responsibilities

This method is restricted to use by, or under the supervision of, analysts experienced in the extraction of aqueous samples for pesticides/PCBs analysis. Each analyst must demonstrate the ability to generate acceptable results with this method. Refer to Katahdin SOP QA-805, current revision, “Personnel Training”.

It is the responsibility of all Katahdin personnel involved in the preparation of aqueous samples for pesticides/PCBs analysis to read and understand this SOP, adhere to the procedures outlined, and to properly document their data in the appropriate lab notebook. Any deviations from the test or irregularities with the

samples should also be recorded in the lab notebook and reported to the Department Manager or designated qualified data reviewer responsible for the data.

It is the responsibility of the Supervisor to oversee that members of their group follow this SOP, that their work is properly documented and to initiate periodic review of the associated logbooks.

1.3 Safety

Users of this procedure must be cognizant of inherent laboratory hazards, proper disposal procedures for contaminated materials and appropriate segregation of hazardous wastes. The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical should be treated as a potential health hazard. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Everyone involved with the procedure must be familiar with the MSDSs for all the materials used in this procedure.

Each qualified analyst or technician must be familiar with Katahdin Analytical Health and Safety Manual including the Katahdin Hazardous Waste Management Plan and must follow appropriate procedures. These include the use of appropriate personal protective equipment (PPE) such as safety glasses, gloves and lab coats when working with chemicals or near an instrument and not taking food or drink into the laboratory. Each analyst should know the location of all safety equipment. Each analyst shall receive a safety orientation from their supervisor, or designee, appropriate for the job functions they will perform.

1.4 Waste Disposal

Wastes generated during the preparation of samples must be disposed of in accordance with the procedures described in the current revision of the Katahdin Hazardous Waste Management Plan and Safety Manual and SOP SD-903, “Sample Disposal,” current revision. Expired standards are lab packed, placed in the Katahdin hazardous waste storage area, and disposed of in accordance with this SOP.

Any methylene chloride solvent waste generated during the rinsing of glassware etc. should be disposed of in the “D” waste stream satellite accumulation area nearest the point of generation. This includes the methylene chloride waste layer generated during CLLE extraction. Special care should be taken to pour this layer off into the appropriate waste stream, leaving the sample waste to be disposed of as follows. Since Pesticide/PCB samples are at a neutral pH, SEP funnel or CLLE sample waste may be dumped into either the “N-Hi” or “N-low” satellite accumulation area. Acetone and hexane are considered flammable waste, and should be disposed of in the “O” waste stream satellite accumulation area nearest the point of generation. Acid waste generated during the cleanup of PCB samples should be disposed of in the “O” satellite accumulation area nearest the point of generation. Please refer to

the current revision of SOP CA-107 for the location of satellite waste accumulation areas

2.0 SUMMARY OF METHOD

Pesticides/PCBs are extracted from aqueous samples using methylene chloride and separatory funnel, continuous liquid-liquid apparatus or Automated Extractor System (SPE), following EPA Methods 3510, 3520, 3535A and EPA Method 608. The methylene chloride is exchanged with hexane for the final extract. Method detection limit studies must be performed annually for pesticides/PCBs using all extraction methods, if the extraction lab wishes to use either or all techniques. Method 3510 (separatory funnel) is generally preferred for pesticides/PCBs since organochlorine pesticides may dechlorinate if under elevated pH conditions for an extended period of time. (Section 3.2, Method 3510B, Rev. 2, 9/94)

3.0 INTERFERENCES

Solvents, reagents, glassware, and other sample preparation apparatus may yield interferences to GC analysis due to the presence of contaminants. These contaminants can lead to discrete artifacts or elevated baselines in chromatograms. Routinely, all of these materials must be demonstrated to be free from interferences under the conditions of the analysis by running reagent blanks. Interferences caused by phthalate esters can pose a major problem in pesticide analysis. Common flexible plastics contain varying amounts of phthalates which are easily extracted during laboratory operations, so cross-contamination of glassware frequently occurs when plastics are handled. Interferences from phthalates can best be minimized by avoiding the use of such plastics in the laboratory. At no time may gloves which have not been tested for phthalates or gloves known to contain phthalates be used or stored in the organic extraction lab. Additionally, whenever possible plastic items in this lab must be replaced with metal or Teflon or other non-phthalate plastic substitute.

Special care should be taken to ensure clean glassware and apparatus are used, pre-rinsed with the appropriate solvent prior to use. Solvents should be analyzed prior to use to demonstrate that each lot is free of contaminants that may interfere with the analysis.

Interferences coextracted from the samples will vary considerably from source to source. If analysis of an extracted sample is prevented due to inteferences, further cleanup of the sample extract may be needed to minimize interferences.

4.0 APPARATUS AND MATERIALS

Prior to use, all glassware must be rinsed three times with the solvent to be used for extraction.

4.1 Separatory Funnel - 2000 mL capacity, Nalgene Teflon FEP separatory funnels with Nalgene Tefzel® screw-cap closures (or equivalent)

4.2 Concentrator tube - 10 mL, graduated

4.3 Evaporative flask - Kuderna-Danish, 500 mL capacity attached to concentrator with neck clips

4.4 Snyder column - Kuderna-Danish, three ball macro

4.5 Graduated cylinders - 100 mL, 1000 mL, or 2000 mL

4.6 Short Stem Funnels

4.7 250 mL amber collection bottles with Teflon-lined caps

4.8 12 mL and/or 16 mL glass vials with Teflon-lined caps

4.9 Continuous liquid-liquid extractors (CLLE) including body, 500 mL flat bottom boiling flask and Alhin condensers

4.10 Filter paper, 18.5 cm, Fisherbrand or Whatman 114V (or equivalent)

4.11 Nitrogen evaporation apparatus.

4.12 Boiling chips - approximately 10/40 mesh, Teflon or selenized carborundum, 12 mesh (or equivalent). Cleaned by Soxhlet.

4.13 Water bath - eight position concentric ring bath or equivalent, equipped with a calibrated thermometer.

4.14 Vials, 60 mL with PTFE – lined screw caps.

4.15 Horizon SPE-DEX 4790 Automated Extractor System.

4.16 Atlantic DVB disks, or equivalent.

4.17 1-L amber bottles

5.0 REAGENTS

5.1 Laboratory reagent grade water - water in which an interferent is not observed at or above the PQL for any parameter of interest (carbon filtered ASTM Type II water or equivalent)

5.2 Sodium Hydroxide (10N) – Purchased from vendor, “Baker-analyzed”, or equivalent

5.3 Sodium Sulfate (ACS) - Granular, anhydrous. Bake at 400°C for 4 hours (may be done by vendor). Purify by rinsing three times with pesticide grade methylene chloride. Allow residual methylene chloride to evaporate before use. Stored in a Teflon capped glass bottle.

5.4 Sulfuric Acid Solution (1:1) - Add 500 mL concentrated sulfuric acid (certified ASC grade or better) slowly to 500 mL laboratory reagent grade water. Prepare as needed and store in a ground glass stoppered bottle.

5.5 Methylene Chloride ($MeCL_2$) - Pesticide grade or better. Lot must be verified by concentrating 300-400 mL to 1.0 mL and evaluating by GC/MS.

5.6 Acetone and Hexane - Pesticide grade or better. Lot must be verified by concentrating approximately 20-30 mL to 1.0 mL and evaluating by GC/ECD.

5.7 Pesticide/PCB Surrogate spiking solution - Prepare a solution of decachlorbiphenyl (DCB) and tetrachloro-meta-xylene (TCMX) at a concentration of 1.0 ug/mL ea in acetone. Store the solution at –10 to -20 °C in a Teflon sealed container. Solution must be verified by GC/ECD prior to use, and must be replaced every 6 months or sooner if degradation is evident.

5.8 Pesticide Matrix Spike/Lab Control Sample spiking solution - Prepare a matrix spiking solution in pesticide grade methanol that contains all target analytes listed below:

ANALYTE	ug/mL
4,4’-DDT	0.5
4,4’-DDD	0.5
4,4’-DDE	0.5
Aldrin	0.5
Dieldrin	0.5
Endrin	0.5
Endrin Aldehyde	0.5
Endrin Ketone	0.5
Endosulfan I	0.5
Endosulfan II	0.5
Endosulfan Sulfate	0.5
alpha-BHC	0.5

ANALYTE (cont.)	ug/mL
beta-BHC	0.5
delta-BHC	0.5
gamma-BHC (Lindane)	0.5
Heptachlor	0.5
Heptachlor epoxide	0.5
Methoxychlor	0.5
alpha-Chlordane	0.5
gamma-Chlordane	0.5

5.9 PCB Matrix Spike/Lab Control Sample spiking solution - Prepare a matrix spiking solution in pesticide grade acetone that contains 5.0ug/ml ea of Aroclor® 1016/1260 mix (Restek catalog# 32039).

5.10 Store the spiking solutions at –10 to -20 °C in a Teflon sealed container. The solutions must be verified by GC/ECD prior to use, and must be replaced every 6 months or sooner if degradation is evident.

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

Samples are collected in 1 L amber bottles and held at 4 (±2) °C until time of extraction.

Holding time for extraction of aqueous samples for Methods 3510, 3520, and 3535 is 7 days from date of sample collection, although the analyst should be aware that actual holding times employed might be project/program specific.

7.0 PROCEDURES

The following information must be recorded in the extraction logbook.

- Extraction method
- Surrogate and spike IDs
- Lot numbers of all solvents, acids and bases, sodium sulfate, filter paper
- Nitrogen evaporation water bath temperature
- Sample pH if appicable
- Extraction and Concentration dates
- Extraction and Concentration analyst
- Sample ID or QC sample ID
- Initial and final volumes or weight
- Surrogate and spike amounts
- Any sample cleanup preformed
- Final extract tray location
- Any comments regarding the sample extraction (ie. Emulsion)

- Prep batch start time and end time
- CLLE start time and end time
- Lot number of the vials the concentrated extracts are stored in.

SEPARATORY FUNNEL SAMPLE EXTRACTION

If an emulsion prevents acceptable recovery or client history indicates samples may demonstrate matrix interence, then samples should be extracted by continuous liquid-liquid extraction (CLLE).

7.1 Rinse all glassware three times with methylene chloride prior to use.

7.2 Label a 2 L Teflon separatory funnel and a 250 mL amber collection bottle clearly. Label should include laboratory sample number, matrix, analyte, and extraction date. Be sure that the detachable stopcocks are secured to the separatory funnels before adding samples.

7.3 Measure the initial volume by comparing the meniscus of the sample with the reference bottle of the same bottle type. Please refer to SOP CA-108, "Basic Laboratory Technique", for the reference bottle verification procedure. Record the volume and any notable characteristics (e.g. color, presence of sediment, or odor) in the extraction logbook.

7.4 Transfer the contents of the sample bottle to a 2 L separatory funnel.

7.5 Transfer 1 L of laboratory reagent grade water to a 2 L separatory funnel. This serves as a method blank for the extraction batch. A method blank must be prepared for every daily extraction batch of twenty or fewer samples.

7.6 Transfer 1 L of laboratory reagent grade water to a 2 L separatory funnel for each analysis to be performed (pesticide and/or PCB). This will serve as a Laboratory Control Sample (LCS). When Pesticides and PCBs are extracted together, a LCS and LCSD set must be extracted for each analysis. An LCS is required for every daily extraction batch of twenty or fewer samples and each analysis. If an MS/MSD pair is not extracted on a particular day, an LCS/LCSD pair may be required in order to meet client-specific or program-specific requirements. This information will be disseminated from the project manager or Department Manager.

7.7 A matrix spike/matrix spike duplicate (MS/MSD) is to be prepared as requested by a client or, at a minimum, one pair per 20 samples or every 14 days and each analysis(refer to the logbook page, "date QC expires"). Transfer two additional 1 L aliquots of sample to 2 L separatory funnels for a matrix spike and matrix spike duplicate (MS/MSD) for each analysis. When Pesticides and PCBs are extracted together, a MS and MSD set must be extracted for each analysis. Note: Sufficient sample volume should be available without depleting all remaining sample aliquots.

7.8 Check the pH of the samples. If it is not between pH 5 and 9, adjust the pH with 10N sodium hydroxide or 1:1 sulfuric acid solution. Note the addition of NaOH or H_2SO_4 in the extraction logbook.

7.9 Using a gas-tight syringe, add 1.0 mL of surrogate spiking solution to all samples the blank, LCS/LCSD(s) and MS/MSD(s), if performed.

7.10 Using a gas-tight syringe, add 1.0 mL of pesticide or PCB matrix spiking solution to the appropriate LCS, LCSD, MS and MSD if performed.

7.11 To each empty sample bottle add 60 mLs of methylene chloride, rinse the bottle and transfer the solvent into the appropriate separatory funnel. Add 60 mL of methylene chloride directly to the blank and LCS/LCSD(s).

7.12 Ensure that each screw cap is secured tightly to the separatory funnel to prevent leaks. Shake briefly and vent in hood to release pressure. Extract the sample by shaking the funnel on mechanical shaker for 3 minutes. Allow phases to separate for at least 10 minutes. Drain the methylene chloride layer into the 250 mL amber collection bottle.

7.13 If an emulsion forms, mechanical techniques must be employed to achieve maximum separation and solvent recovery. Such means include swirling and centrifugation and draining through a small separatory funnel. In certain instances, transferring the entire sample into a continuous liquid-liquid extractor may be the only alternative. If any such techniques are used, they must be noted in the extractions logbook.

7.14 Add a second 60 mL aliquot of methylene chloride to the separatory funnel and extract for the second time (see 7.10 - 7.12). Collect the methylene chloride layer in the same 250 mL amber collection bottle.

7.15 Repeat the extraction for a third time as described in 7.13.

7.16 Proceed to Section 7.53 for extract concentration procedures.

CONTINUOUS LIQUID-LIQUID SAMPLE EXTRACTION (CLLE)

7.17 Set up the CLLE apparatus. All glassware should be rinsed three times with methylene chloride and the extract flasks properly labeled.

7.18 Add 2-3 boiling stones to the round bottom flask and approximately 500 - 600 mL of methylene chloride to the CLLE body.

7.19 Add 1 L laboratory reagent grade water to a CLLE body. This is the method blank for this extraction batch. Be sure that no water leaks into the round bottom flask. A method blank is required for every extraction batch of twenty or fewer samples.

7.20 Mark the sample level (meniscus) on the sample bottle with a wax crayon so that the volume can be measured (this may be done prior to removal from the walk-in cooler). Transfer the sample to a CLLE body, being sure that no water leaks into the round bottom flask.

7.21 Prepare an LCS for every daily extraction batch of twenty or fewer samples and each analysis (pesticide and/or PCB). Add 1 L of laboratory reagent grade water to a CLLE body. If an MS/MSD pair is not extracted on a particular day, an LCS/LCSD pair may be required in order to meet client-specific or program-specific requirements. This information will be disseminated from the project manager or Department Manager. When Pesticides and PCBs are extracted together, a LCS and LCSD set must be extracted for each analysis.

7.22 Mark the sample levels on the sample bottles. Transfer the samples to the CLLE bodies.

7.23 Check the pH of the samples. If it is not between pH 5 and 9, adjust the pH with 10N sodium hydroxide or 1:1 sulfuric acid solution. Note the addition of NaOH or H_2SO_4 in the extraction logbook.

7.24 Transfer two 1 L portions of a sample to CLLE bodies for each analysis for preparation of a matrix spike/matrix spike duplicate if required. An MS/MSD is required if requested by the client or per 20 samples, whichever occurs first. When Pesticides and PCBs are extracted together, a MS and MSD set must be extracted for each analysis. Note: Sufficient sample volume should be available without depleting all remaining sample aliquots.

7.25 For each sample, rinse the original sample container with approximately 30 mL of methylene chloride. Add this rinse to the CLLE body.

7.26 Determine the initial volume of the samples by comparing the grease marking where the sample meniscus was to the reference bottle located in the lab. Please refer to SOP CA-108, “Basic Laboratory Technique”, for the reference bottle verification procedure. Record the volume and any notable characteristics (e.g. color, presence of sediment, or odor) in the extraction logbook.

7.27 Add 1.0 mL of the Pesticide/PCB Surrogate Spike to each sample including the blank, LCS/LCSD and MS/MSD, if performed.

7.28 Add 1.0 mL of Pesticide or PCB Matrix Spike to the appropriate LCS/LCSD and MS/MSD pair, if performed, and stir.

7.29 Attach cooling water Allihn condensers, after first rinsing each 45/50 joint with methylene chloride. Turn on the heating mantles and allow the samples to extract for at least 18 hours, total extract time may go up to 20 hours. Turn off the mantles and let samples cool.

7.30 Proceed to Section 7. 53 for sample extract concentration procedures.

EXTRACTION WITH AUTOMATED EXTRACTOR SYSTEM (SPE)

Alternatively, samples may be extracted using the Horizon Automated Extractor System (Figure 2)

Purging the Extractor Vessels

7.31 Check and fill all necessary solvent bottles (acetone, laboratory reagent grade water and hexane) as needed. Check and empty the two waste containers as needed.

7.32 Turn on nitrogen tank to 60 psi. Turn the instrument pressure on top of the controller to 50 psi. Turn the solvent bottle pressure to 10 psi.

7.33 Turn on the Horizon controller (switch in the back).

7.34 Check the lubrication oil on the air pump. Fill as needed. Turn the air pump on.

7.35 Clean the glass sensors that are located on the back of the dispensing stems of the extractors using a Kim Wipe. This is to remove any residue that may interfere with the sensors.

7.36 Attach 19/22 adapters to 40-mL vials and attach beneath the disk holder platforms of the extractors. Assembly per owner's manual and place empty Horizon disk holder assemblies on top of the disk holder platforms. There should be roughly 1 cm separating the speedisk from the extractor downtube.

7.37 Check to be sure that all extractors have empty sample bottles loaded on top. If not, use a Horizon cap on a one liter empty bottle and firmly place the bottlenose down into the extractor.

7.38 Press *select* on the control panel to designate an extractor (1, 2, 3, 4 or "." for all), then press *enter*.

7.39 Type 8081.9, and press enter to select pesticide/PCB purge method. Once the method is loaded, start the extractors by pressing the *start* buttons on the individual extractors. The red LED will blink when the method is complete.

7.40 Repeat this process 2-3 times before using the Horizon autoextractors.

ANALYSIS OF SAMPLES WITH AUTOEXTRACTOR

7.41 Label a 60 ml vial with the sample to be extracted. Attach 19/22 adapter to vial and attach beneath the disk holder platforms of the extractors.

7.42 Place an Atlantic DVB disk (or equivalent) into a Horizon disk holder assembly and assemble per owner's manual. Place the disk holder assemble on top of the disk holder platform. There should be roughly 1 cm separating the speedisk from the extractor downtube

7.43 Mark the volume level of liquid in each sample on the outside using a grease pencil.

7.44 Add 1 L laboratory reagent grade water to 1 L amber bottle. This is the method blank for this batch. A method blank is required for every extraction batch of twenty or fewer samples.

7.45 Prepare an LCS for every daily extraction batch of twenty or fewer samples and each analysis, pesticide and/or PCB. Add 1 L of laboratory reagent grade water to a 1 L amber bottle. If an MS/MSD pair is not extracted on a particular day, an LCS/LCSD pair may be required to meet client specific or program specific requirements. This information will be disseminated from the project manager or department manager.

7.46 Add 1.0 mL of Pesticide/PCB surrogate spike to each sample including the blank, LCS/LCSD and MS/MSD, if required. Recap samples and shake well.

7.47 Add 1.0 mL of pesticide or PCB matrix spike to the appropriate LCS/LCSD and MS/MSD samples. Recap and shake well.

7.48 Remove cap and add 5.0 mL of 1:1 H_2SO_4 to each sample including the blank, LCS/LCSD and MS/MSD set immediately prior to extracting the sample.

7.49 Remove the cap from each sample bottle and cover with tin foil. Screw a Horizon adapter cap over the tin foil. Invert the bottle and check for leaks.

7.50 Load the sample bottle on the holder and twist ¾ of a rotation. Stop twisting when air bubbles rise to the top of the sample bottle. Do not twist completely around. The foil may loosen and jam the valve.

7.51 Press *select* on the control panel to designate an extractor (1, 2 or "." for both), then press *enter*.

7.52 Type in 8081.3 for the method and press enter. Once the method is loaded, start the extractors by pressing the *start* buttons on the individual extractors. The red LED will blink when the method is complete. The extract will be collected in the 60 ml vial.

7.53 Sample is now ready to reduce to 10 mL final volume.

NOTE: The instrument methods stated above apply specifically to the Atlantic DVB disk. Instrument methods may need to be modified with the usage of different filters and/or to increase recoveries. See instrument logbook for current methods in use.

CONCENTRATION OF WATER SAMPLE EXTRACTS

7.54 Rinse the K-D glassware (flask, concentration tube, funnel and Snyder column, including the ground glass joints on the flask and columns) three times with methylene chloride (or hexane for samples extracted with the Autoextractor) before assembling. Add two boiling chips to the K-D. Insert fluted 18.5 cm filter papers into short stem powder funnels and add ~ 2 inches of sodium sulfate crystals. Rinse the sodium sulfate in the assembled funnels with ~20-30 mLs of methylene chloride (hexane for samples extracted with the Autoextractor). Place the assembled K-D's under the funnels.

7.55 For methylene chloride extracts, add approximately 50 mL Hexane to funnel and let drain through. Since methylene chloride has a lower boiling point than Hexane, this will result in a final extract in hexane only.

7.56 Transfer the methylene chloride or hexane extracts to the K-D concentrator setups through the sodium sulfate in the funnels. This is the drying step, which is required to remove residual water from the extracts. Any large water layers must be removed by other means, prior to pouring through the sodium sulfate. After pouring all of the extract volume through the sodium sulfate, rinse the extract bottle three times with ~ 2 – 3 mLs of methylene chloride(or hexane for samples extracted with the Autoextractor). Add the rinsings through the sodium sulfate to complete a quantitative transfer. Rinse the sodium sulfate with ~ 15 mLs of methylene chloride (or hexane for samples extracted with the Autoextractor) and allow to drain.

7.57 Transfer the labels from the collection bottles or round bottom flasks (from the CLLE extraction) to the K-Ds. Remove the funnel and attach a 3-ball macro Snyder column. Pre-wet the Snyder column with 1 mL of methylene chloride (or hexane for samples extracted with the Autoextractor).

7.58 Place the K-D in a hot water bath (85-90°C). Gently swirl K-D in the water until boiling begins. At the proper distillation rate, the Snyder balls should chatter but the chambers should not flood with condensed solvent. The K-D should be kept in a vertical orientation while on the bath. When the apparent volume in the concentrator tube reaches ≈ 5-6 mL, remove the K-D from the water bath. Allow the K-D to cool for 10 minutes. Rinse the Snyder column lower joint with ≈ 1 mL of hexane. Remove the Snyder column. Wipe off any water from the neck above the lower joint of the flask. Separate the K-D flask from the concentrator tube, rinsing the ground glass joint with ≈ 1 mL hexane.

7.59 Reduce the extracts to ≈ 1 mL using Nitrogen blow-down apparatus. The bath temperature must be no higher than the boiling point of the solvent (45 °C for hexane). Turn the gas to 3 psi. Be careful not to splash the extract out of the tube. During concentration on the N-evap, the internal wall of the concentrator tube and the N-evap sparging pipet must be rinsed down at least once or twice with ≈1 mL of methylene chloride. The solvent level in the concentrator tube must be positioned below the level of the water bath as much as possible to prevent water from condensing into the sample extract. As the extract volume is reduced, lower the N_2 sparging needle closer to the surface of the extract to expedite the concentration. Note any problems or extract losses, if they occur, in the extractions logbook. Transfer extract to a 12 or 16 mL vial. Using a reference vial for volume comparison, adjust the final extract volume to 10 mL by rinsing sides of tube with hexane and transferring rinsings to vial.

7.60 If at any point in the concentration procedure the concentrator tube goes dry – reextract the sample immediately.

7.61 Transfer the label from the concentrator tube to the vial. Store extract vials at a temperature of 4 ± 2 °C until ready for analysis. Indicate in the extractions logbook the box number and “tray location” of the individual extract vials.

7.62 All sample extracts for 8082 PCB analysis must undergo a sulfuric acid wash (cleanup) prior to analysis. All sample extracts for 8081 pesticide analysis do not undergo further cleanup unless requested by the client. Therefore, all sample extracts for combined 8081/8082 analyses must be split. Prior to splitting, mix contents of vial well. One portion must be acid cleaned for 8082 analysis. The associated method blank must be split and acid-cleaned in the same fashion. PCB LCSs and matrix spikes are acid cleaned also. Pesticide LCSs and matrix spikes are not subjected to further cleanup. Please refer to Katahdin SOP CA525 (current revision), Extract Cleanup Using Sulfuric Acid, for further instructions.

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

Each extractions analyst must demonstrate proficiency in performing the extractions that prepare samples for analysis. Demonstration consists of preparation (extraction), by the analyst, of at least four aliquots which are then analyzed according to the analytical method in question. These QC samples must meet all quality control acceptance limits. Demonstration must be documented by use of a form which summarizes the results of the analysis of these aliquots, calculated percent recoveries, and standard deviation. Demonstration of proficiency must be done one time per analyst initially and then annually thereafter. Refer to SOPs QA-805 and QA-807, current revision.

If, upon analysis of the extracted samples, it is discovered that quality control acceptance criteria have not been met, all associated samples must be evaluated against all the QC. In some cases data may be reported, perhaps with narration, while in other cases, other

corrective action may be taken. The corrective actions may include re-extraction of the samples associated with the quality control sample that did not meet acceptance criteria, or may include making new reagents and standards if the standardization is suspect. These decisions are based on holding time considerations, client and project specific Data Quality Objectives and on review of chromatograms. The supervisor, Operations Manager, and/or Quality Assurance Officer may be consulted to evaluate data. Some samples may not be able to be reanalyzed within hold time. In these cases "qualified" data with narration may be advisable after consultation with the client.

Much of the work performed at the lab is analyzed in accordance with specific QC requirements spelled out in a project specific Quality Assurance Project Plan (QAPP) or in a program specific Quality Systems Manual (QSM). The reporting limits, acceptance criteria and/or corrective actions may be different than those specified in this SOP. In these cases the appropriate information will be communicated to the Department Manager and/or senior chemists before initiation of the analyses so that specific product codes can be produced for the project. In addition, the work order notes for each project will describe the specific QAPP or QSM to be followed.

A method blank must be extracted for each and every item listed below:

- Each day of extraction (24 hours midnight - midnight)
- Each extraction method
- Every 20 samples extracted in a 24-hour period

A laboratory control sample (LCS) is required for each and every item listed below:

- Each extraction method
- Every extraction batch of twenty or fewer samples
- Each analysis (pesticide and/or PCB) to be performed

Refer to the current revision of the applicable Katahdin SOP for analysis of Pesticides and PCBs for quality control acceptance criteria.

9.0 METHOD PERFORMANCE

Refer to the applicable analytical SOP.

10.0 APPLICABLE DOCUMENTS/REFERENCES

Test Methods for Evaluating Solid Waste - Physical/Chemical Methods, Methods 3510C and 3520C, USEPA SW-846, Third Edition, Final Update III, December 1996.

40 CFR 136, Appendix A, "Test Procedures for Analysis of Organic Pollutants," Method 608, June, 1998 edition.

TITLE: PREPARATION OF AQUEOUS SAMPLES FOR PESTICIDES/PCBs ANALYSIS

LIST OF TABLES AND FIGURES

TITLE: PREPARATION OF AQUEOUS SAMPLES FOR PESTICIDES/PCBs ANALYSIS

TABLE 1

SUMMARY OF METHOD MODIFICATIONS (METHOD 3510, current revision)

TOPIC	KATAHDIN SOP CA-515-06	METHOD 3510, current revision
Apparatus/Materials	1. 12 or 16 mL vials used for final extract 2. 250 mL amber bottle or flask used 3. 1.0 mL syringe 4. short stem funnels	1. 2 mL vials used for final extract 2. 250 mL Erlenmeyer flask 3. 5.0 mL syringe 4. drying column
Reagents		
Sample preservation/ handling	1. entire contents of 1 L sample bottle transferred to separatory funnel	1. one liter graduated cylinders used to transfer initial sample volume to separatory funnel
Procedures	1. extract collection in amber bottle or Erlenmeyer flask 2. extract dried using Na_2SO_4 in short stem funnels 3. no apparatus height specification for concentration on water bath 4. sample removed from water bath when volume reaches ~10 mL 5. hexane added directly to K-D body at start of concentration process	1. extract collection in Erlenmeyer flask 2. extract dried using Na_2SO_4 in drying columns 3. partially immerse concentrator tube in water and lower apparatus to complete concentration in 10-15min 4. sample removed from water bath when volume reaches 1-2 mL 5. solvent exchange via large K-D with addition of 50 mL hexane after concentrating methylene chloride extract to 1 mL
QC - Spikes		
QC - LCS		
QC - Accuracy/Precision		
QC - MDL		

TITLE: PREPARATION OF AQUEOUS SAMPLES FOR PESTICIDES/PCBs ANALYSIS

TABLE 2

SUMMARY OF METHOD MODIFICATIONS (METHOD 3520, current revision)

TOPIC	KATAHDIN SOP CA-515-06	METHOD 3520, current revision
Apparatus/Materials	1. short-stem funnels 2. 12 or 16 mL vials used for final extract	1. drying columns 2. 2 mL vials used for final extract
Reagents		
Sample preservation/ handling	1. entire contents of 1 L sample bottle transferred to CLLE	1. one liter graduated cylinders used to tranfer initial sample volume to CLLE
Procedures	1. CLLE for 18 $\pm$ 2 hours 2. extract dried using Na_2SO_4 in short stem funnels 3. no apparatus height specification for concentration on water bath 4. sample removed from water bath when volume reaches ~10 mL 5. hexane added directly to K-D body at start of concentration process	1. CLLE for 18-24 hours 2. extract dried using Na_2SO_4 in drying columns 3. partially immerse concentrator tube in water and lower apparatus to complete concentration in 10-15min 4. sample removed from water bath when volume reaches 1-2 mL 5. solvent exchange via macro K-D with addition of 50 mL hexane after concentrating methylene chloride extract to 1 mL
QC - Spikes		
QC - LCS		
QC - Accuracy/Precision		
QC - MDL		

TITLE: PREPARATION OF AQUEOUS SAMPLES FOR PESTICIDES/PCBs ANALYSIS

TABLE 3

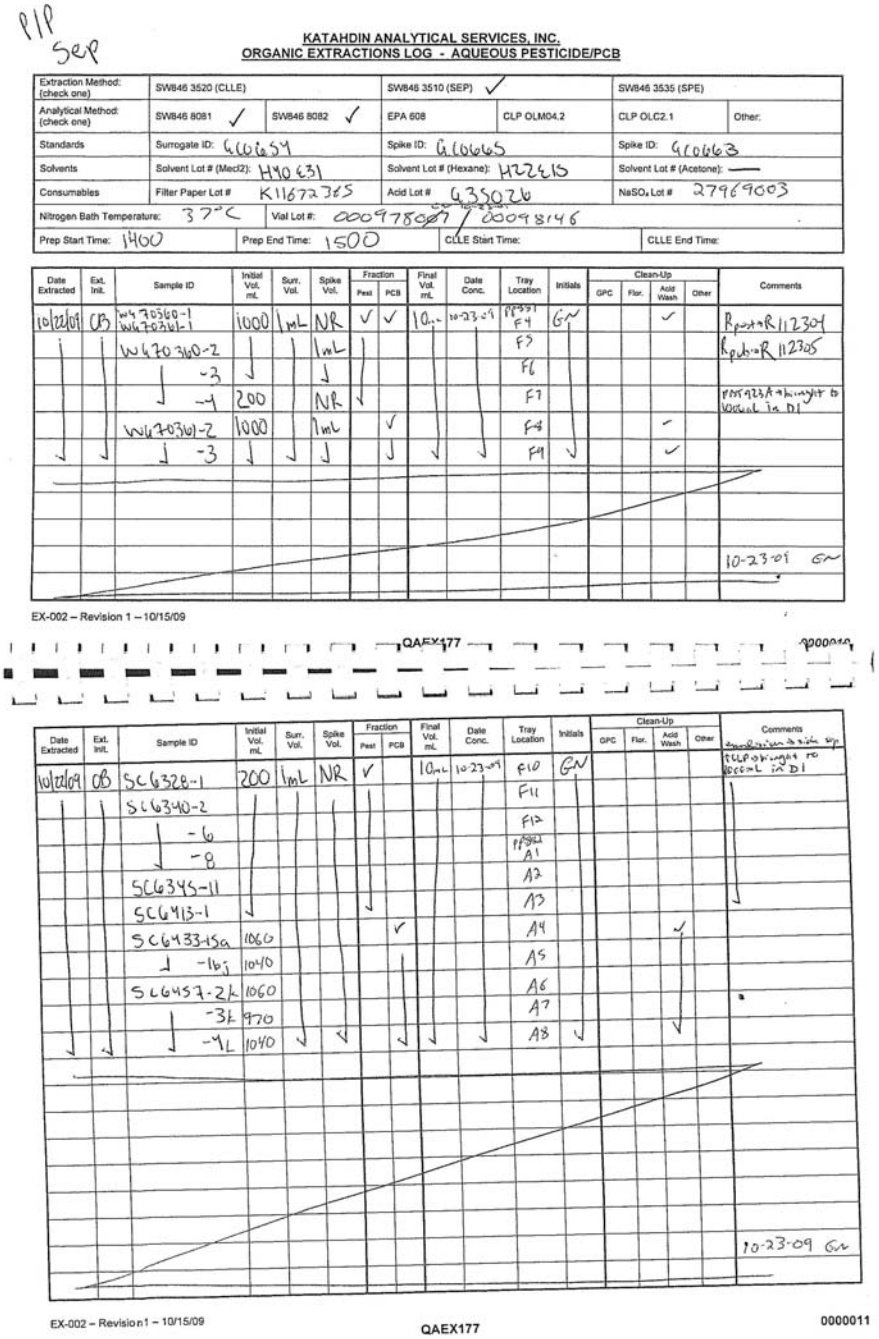
SUMMARY OF METHOD MODIFICATIONS (METHOD 3535, current revision)

TOPIC	KATAHDIN SOP CA-515-06	METHOD 3520, current revision
Apparatus/Materials	1. Horizon SPE-DEX 4790 Automated Extractor System.	1. Empore solid-phase extraction system
Reagents		
Sample preservation/ handling	1. entire contents of 1 L sample bottle transferred to separatory funnel	1. one liter graduated cylinders used to transfer initial sample volume to separatory funnel
Procedures	1. no methanol addition 2. extraction using Horizon SPE-DEX 4790 Automated Extractor System. 3. extract dried using Na_2SO_4 in short stem funnels 4. no apparatus height specification for concentration on water bath 5. sample removed from water bath when volume reaches ~10 mL 6. hexane added directly to K-D body at start of concentration process	1. 5mL methanol added to all samples and blanks 2. extraction using Empore solid-phase extraction system 3. extract dried using Na_2SO_4 in drying columns 4. partially immerse concentrator tube in water and lower apparatus to complete concentration in 10-15min 5. sample removed from water bath when volume reaches 1mL 6. solvent exchange via macro K-D with addition of 50 mL hexane after concentrating methylene chloride extract to 1 mL
QC - Spikes		
QC - LCS		
QC - Accuracy/Precision		
QC - MDL		

TITLE: PREPARATION OF AQUEOUS SAMPLES FOR PESTICIDES/PCBs ANALYSIS

FIGURE 1

EXAMPLE OF LOGBOOK PAGE



PIP Sep

KATAHDIN ANALYTICAL SERVICES, INC.
ORGANIC EXTRACTIONS LOG - AQUEOUS PESTICIDE/PCB

Extraction Method: (check one)	SW846 3520 (CLLE)		SW846 3510 (SEP) ✓		SW846 3535 (SPE)		
Analytical Method: (check one)	SW846 8081 ✓	SW846 8082 ✓	EPA 608	CLP OLM04.2	CLP OLC2.1	Other:	
Standards	Surrogate ID: G606654		Spike ID: G606665		Spike ID: G606663		
Solvents	Solvent Lot # (MeCl2): H90631		Solvent Lot # (Hexane): H226L5		Solvent Lot # (Acetone): —		
Consumables	Filter Paper Lot # K11672365		Acid Lot # G35026		$NaSO_4$ Lot # 27969003		
Nitrogen Bath Temperature: 37°C		Vial Lot #: 000978087 / 00098146					
Prep Start Time: 1400	Prep End Time: 1500		CLLE Start Time:		CLLE End Time:		

Date Extracted	Ext. Init.	Sample ID	Initial Vol. mL	Surr. Vol.	Spike Vol.	Fraction: Pest	Fraction: PCB	Final Vol. mL	Date Conc.	Tray Location	Initials	Clean-Up: GPC	Clean-Up: Flor.	Clean-Up: Acid Wash	Clean-Up: Other	Comments
10/22/09	OB	W670360-1 W670361-1	1000	1mL	NR	✓	✓	10mL	10-23-09	F4	GN			✓		Repeat of R112304
↓	↓	W670360-2	↓	↓	1mL	↓		↓	↓	F5	↓					Repeat of R112305
		↓ -3	↓	↓	↓					F6						
		↓ -4	200		NR	✓				F7						PFK923A weight to volume in DI
		W670361-2	1000		1mL		✓			F8				✓		
↓	↓	↓ -3	↓	↓	↓		↓	↓	↓	F9	↓			✓		
																10-23-09 GN

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Date Extracted	Ext. Init.	Sample ID	Initial Vol. mL	Surr. Vol.	Spike Vol.	Fraction: Pest	Fraction: PCB	Final Vol. mL	Date Conc.	Tray Location	Initials	Clean-Up: GPC	Clean-Up: Flor.	Clean-Up: Acid Wash	Clean-Up: Other	Comments
10/22/09	OB	SC6328-1	200	1mL	NR	✓		10mL	10-23-09	F10	GN					TCLP extract weight to volume in DI
↓	↓	SC6340-2	↓	↓	↓	↓		↓	↓	F11	↓					↓
		↓ -6								F12						
		↓ -8								A1						
		SC6345-11								A2						
		SC6413-1	↓			↓				A3						
		SC6433-15a	1060				✓			A4				✓		
		↓ -16j	1040				↓			A5						
		SC6457-2L	1060							A6						
		↓ -3L	970							A7						
↓	↓	↓ -4L	1040	↓	↓		↓	↓	↓	A8	↓			↓		
																10-23-09 GN

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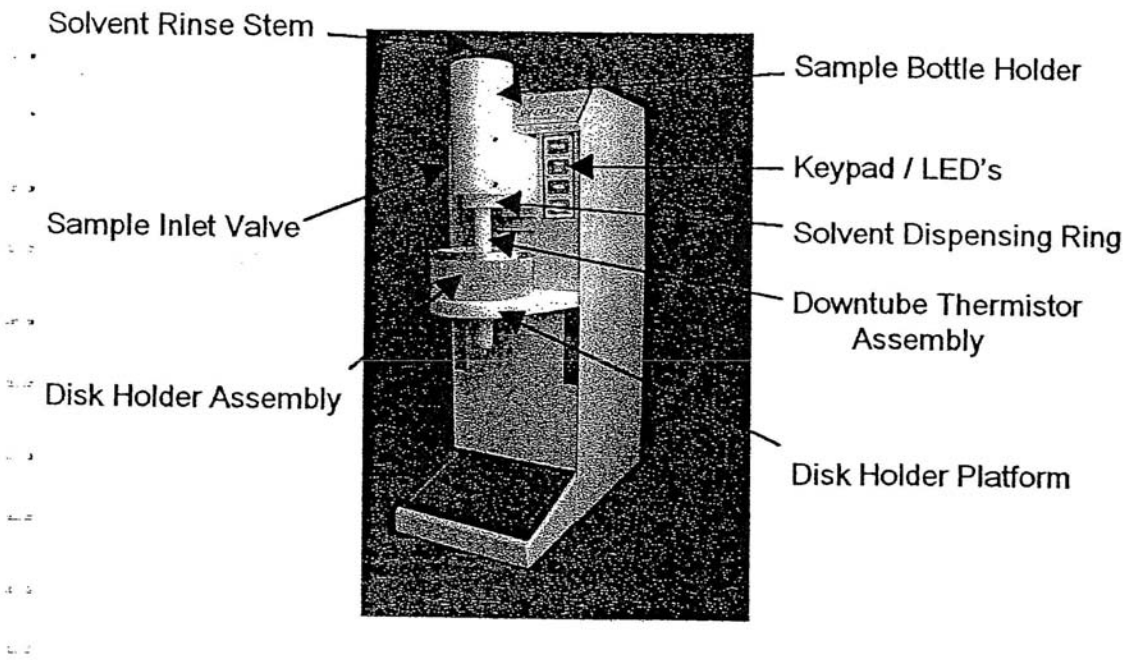
0000011

FIGURE 2

HORIZON AUTOEXTRACTOR SYSTEM DIAGRAM



TITLE: PREPARATION OF SEDIMENT/SOIL SAMPLES BY SOXHLET EXTRACTION USING METHOD 3540 FOR PESTICIDE/PCB ANALYSIS

Prepared By: Mike Thomas Date: 7/98

Approved By:

Group Supervisor: Michael F. Thomas Date: 11/15/00

Operations Manager: [signature] Date: 11/15/00

QA Officer: Deborah J. Nadeau Date: 11·15·00

General Manager: [signature] Date: 11/16/00

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
01	Minor changes throughout. Clarifications to procedure section.	DN	11·15·00	11/15/00
02	Added definitions to section 1.1. Wording changed or added to clarify sections 5, 6, 8, + 9. New figure	MRC	11.08.04	11.08.04
03	Sect. 7.1.2 - adding the step to rinse forceps also. 7.10 adding condenser temperature and output voltage of variable transformer	LAD	04/06	04/06
04	Added generated waste information. Updated spike list. Added LCSD. Reworded Sect. 7.10 and 7.11 for clarification. Updated Table 1 Replaced figure 1	LAD	09/07	09/07
05	Changed "N-Lo" waste to "K" waste. Updated Logbook example. Sect. 7 - added wording instructing the recording of consumable lot #'s in Logbook.	LAD	07/08	07/08

TITLE: PREPARATION OF SEDIMENT/SOIL SAMPLES BY SOXHLET EXTRACTION USING METHOD 3540 FOR PESTICIDE/PCB ANALYSIS

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
06	Added balance criteria. Changed weight criteria to +/- 0.05g. Minor changes to section 7 to reflect current techniques. Clarified samples being GPC'd are not solvent exchanged into hexane. Updated logbook page. Added CA-108 reference for subsampling information	LAD	08/09	08/09

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SOXHLET EXTRACTION USING METHOD 3540 FOR PESTICIDE/PCB ANALYSIS**

Please acknowledge receipt of this standard operating procedure by signing and dating both of the spaces provided. Return the bottom half of this sheet to the QA Department.

I acknowledge receipt of copy ___ of document **SOP CA-524-06**, titled **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SOXHLET EXTRACTION USING METHOD 3540 FOR PESTICIDE/PCB ANALYSIS.**

Recipient: _____ Date: _____

KATAHDIN ANALYTICAL SERVICES, INC.
STANDARD OPERATING PROCEDURE

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Recipient: _____ Date: _____

1.0 SCOPE AND APPLICATION

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SOXHLET EXTRACTION USING METHOD 3540 FOR PESTICIDE/PCB ANALYSIS**

The purpose of this SOP is to describe the procedure for extracting pesticides/PCBs from solids such as soils, sludges, and wastes using Method 3540. The Soxhlet extraction process ensures intimate contact of the sample matrix with the extraction solvent.

This method is applicable to the isolation and concentration of water-insoluble and slightly water-soluble organics in preparation for a variety of chromatographic procedures including methods 8081 for pesticides and 8082 for PCB's.

1.1 Definitions

METHOD BLANK (laboratory reagent blank): An artificial sample designed to determine if method analytes or other interferences are present in the laboratory environment, the reagents, or the apparatus. For aqueous samples, reagent water is used as a blank matrix; for soil samples, baked organic-free sand is used as a blank matrix. The blank is taken through the appropriate steps of the process.

LABORATORY CONTROL SAMPLE (LCS): A blank that has been spiked with the analyte(s) from an independent source, and is analyzed exactly like a sample. Its purpose is to determine whether the methodology is in control, and whether the laboratory is capable of making accurate and precise measurements. The matrix used should be phase matched with the samples and well characterized.

MATRIX SPIKE/MATRIX SPIKE DUPLICATE (MS/MSD): Predetermined quantities of stock solutions of certain analytes are added to a sample matrix prior to sample extraction and analysis. Samples are split into duplicates, spiked and analyzed. Percent recoveries are calculated for each of the analytes detected. The relative percent difference between the samples is calculated and used to assess analytical precision.

SURROGATES: Organic compounds which are similar to analytes of interest in chemical composition, extraction and chromatography, but which are not normally found in environmental samples. These compounds are spiked into all blanks, standards, samples and spiked samples prior to analysis. Percent recoveries are calculated for each surrogate.

1.2 Responsibilities

This method is restricted to use by, or under the supervision of analysts experienced in the extraction of samples for pesticide/PCB analysis. Each analyst must demonstrate and document their ability to generate acceptable results with this method. Refer to Katahdin SOP QA-805, current revision, "Personnel Training and Documentation of Capability".

It is the responsibility of all Katahdin technical personnel involved i n the extraction of

samples for pesticide/PCB analysis to read and understand this SOP, adhere to the procedures outlined, and to properly document their data in the appropriate lab notebook. Any deviations from the test or irregularities with the samples should also be recorded in the lab notebook and reported to the Department Manager or designated qualified data reviewer responsible for this data.

It is the responsibility of the Department Manager to oversee that members of their group follow this SOP, to ensure that their work is properly documented and to indicate periodic review of the associated logbooks.

1.3 Safety

Users of this procedure must be cognizant of inherent laboratory hazards, proper disposal procedures for contaminated materials and appropriate segregation of hazardous wastes. The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical should be treated as a potential health hazard. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Everyone involved with the procedure must be familiar with the MSDSs for all the materials used in this procedure.

Each qualified analyst or technician must be familiar with Katahdin Analytical Health and Safety Manual including the Katahdin Hazardous Waste Plan and must follow appropriate procedures. These include the use of appropriate personal protective equipment (PPE) such as safety glasses, gloves and lab coats when working with chemicals or near an instrument and not taking food or drink into the laboratory. Each analyst should know the location of all safety equipment. Each analyst shall receive a safety orientation from their Department Manager, or designee, appropriate for the job functions they will perform.

1.4 Waste Disposal

Wastes generated during the preparation of samples must be disposed of in accordance with the procedures described in the current revision of the Katahdin Hazardous Waste Plan and Safety Manual and SOP SD-903, "Sample Disposal," current revision. Expired standards are lab packed, placed in the Katahdin hazardous waste storage area, and disposed of in accordance with this SOP.

Any methylene chloride solvent waste generated during the rinsing of glassware etc. should be disposed of in the "D" waste stream satellite accumulation area nearest the point of generation. Acetone and hexane are considered flammable waste, and should be disposed of in the "O" waste stream satellite accumulation area nearest the point of generation. Post-extraction soil samples, used glass wool, and sodium sulfate waste should be disposed of in the soil with organics "I" waste stream satellite accumulation area nearest the point of generation. Acid waste generated

during the cleanup of PCB samples should be disposed of in the "K" satellite accumulation area nearest the point of generation. Please refer to the current revision of SOP CA-107 for the location of satellite waste accumulation areas.

2.0 SUMMARY OF METHOD

2.1 The solid sample is mixed with anhydrous sodium sulfate, placed in a Soxhlet extractor and extracted with methylene chloride.

2.2 The extract is then dried, concentrated, and exchanged into hexane for GC analysis. Sulfuric acid cleanup is performed on extracts for 8082 PCB analysis.

3.0 INTERFERENCES

Solvents, reagents, glassware, and other sample preparation apparatus may yield interferences to GC analysis due to the presence of contaminants. These contaminants can lead to discrete artifacts or elevated baselines in chromatograms. Routinely, all of these materials must be demonstrated to be free from interferences under the conditions of the analysis by running reagent blanks. Interferences caused by phthalate esters can pose a major problem in pesticide analysis. Common flexible plastics contain varying amounts of phthalates that are easily extracted during laboratory operations, so cross-contamination of glassware frequently occurs when plastics are handled. Interferences from phthalates can best be minimized by avoiding the use of such plastics in the laboratory. At no time may gloves that have not been tested for phthalates or gloves known to contain phthalates be used or stored in the organic extraction lab. Additionally, whenever possible plastic items in this lab must be replaced with metal or teflon or other non-phthalate plastic substitute.

Special care should be taken to ensure clean glassware and apparatus are used, pre-rinsed with the appropriate solvent prior to use. Solvents should be analyzed prior to use to demonstrate that each lot is free of contaminants that may interfere with the analysis.

Interferences coextracted from the samples will vary considerably from source to source. If analysis of an extracted sample is prevented due to inteferences, further cleanup of the sample extract may be needed to minimize interferences.

4.0 APPARATUS AND MATERIALS

4.1 a) Soxhlet extractor – 45/50 top joint and 24/40 lower joint.

b) 500 mL flat-bottom boiling flask

c) Allihn cooling water condenser

4.2 Powder Funnels – 100 mm top diameter, 35 mm stem

4.3 Kuderna-Danish (K-D) apparatus

4.3.1 Concentrator tube - 10-mL

4.3.2 Evaporation flask - 500-mL

4.3.3 Snyder column - Three-ball macro

4.4 Nitrogen evaporation (N-EVAP) apparatus.

4.5 Boiling stones, 12 mesh silicon carbide (carborundum) – pre-purified by Soxhlet extraction in methylene chloride

4.6 Water bath - Heated, with concentric ring cover, capable of temperature control (± 5°C). The bath should be used in a hood.

4.7 Vials - Glass, 4, 12, or 16 mL with Teflon-lined screw caps

4.8 Glass wool (fiberglass) - baked at 400°C for a minimum of 4 hours or overnight.

4.9 Heating mantles - Rheostat controlled.

4.10 Disposable glass Pasteur pipets, 5 ¾", and bulbs.

4.11 Drying oven - capable of maintaining 105°C for glassware drying.

4.12 Muffle oven – capable of maintaining 400 °C for baking glass wool and organic-free sand.

4.13 Beakers, 250 or 400 mL

4.14 Top-loading balance - capable of weighing to 0.**01** g.

4.15 Spatulas, stainless-steel

4.16 Long forceps, stainless-steel

4.17 Metal clips – for securing Soxhlets to boiling flasks

4.18 Filter paper, 18.5 cm, Fisherbrand or Whatman 114V (or equivalent)

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SOXHLET EXTRACTION USING METHOD 3540 FOR PESTICIDE/PCB ANALYSIS**

5.0 REAGENTS

5.1 Sodium sulfate (granular, anhydrous and powdered, anhydrous) (ACS reagent grade), Na_2SO_4. Certified by the manufacturer/vendor as purified by heating at 400°C for 4 hours prior to receipt by the laboratory.

5.2 Methylene chloride - (pesticide grade or equivalent) purchased by lot, evaluated prior to use by concentration of 300 mL to 1 mL followed by GC/MS analysis.

5.3 Acetone and hexane – (pesticide grade or equivalent) purchased by lot, evaluated prior to use by concentration of 300 mL to 1 mL followed by GC/MS and GC analysis.

5.4 Organic-free sand, purified by baking at 400 °C at a minimum of 4 hours or overnight. Method blanks serve as checks on the baked sand.

5.5 Surrogate spiking solution - Prepare a solution of decachlorobiphenyl (DCB) and tetrachloro-meta-xylene (TCMX) at a concentration of 1 ug/mL in acetone.

5.6 Matrix Spike/Lab Control Sample spiking solution

5.6.1 Pesticide spike solution – prepare in pesticide grade methanol containing the analytes listed below at concentrations of 0.5 ug/mL.

4,4'-DDD	Endrin
4,4'-DDE	Endrin Aldehyde
4,4'-DDT	Endrin Ketone
Aldrin	gamma-BHC (Lindane)
alpha-BHC	Heptachlor
beta-BHC	Heptachlor Epoxide
delta-BHC	Methoxychlor
Dieldrin	alpha-Chlordane
Endosulfan I	gamma-chlordane
Endosulfan II	Endrin
Endosulfan Sulfate	Endrin Aldehyde

5.6.2 PCB spike solution – prepare Aroclor 1660 (Aroclor 1016 and 1260) in pesticide grade acetone at a concentration of 5.0 ug/mL each.

5.7 Store the solutions mentioned in sections 5.5 and 5.6 at -10 to -20 °C (±2 °C) in a Teflon sealed container. Solution must be verified by GC/ECD prior to use and must be replaced every 6 months or sooner if degradation is evident.

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SOXHLET EXTRACTION USING METHOD 3540 FOR PESTICIDE/PCB ANALYSIS**

Sediment/soil samples must be collected in a soil jar and must be maintained at 4°C (±2°C).

Holding time for extraction of sediment/soil samples for Method 3540 is 14 days from date of sample collection, although the analyst should be aware that actual holding times employed may be project/program specific.

Store all extracts at 4°C (±2°C) in the dark in labeled Teflon-sealed containers. See SOP SD-902, "Sample Receipt and Internal Control," current revision, for storage areas and temperature maintenance procedures.

7.0 PROCEDURES

7.1 Preparing the Soxhlet Extraction Apparatus

7.1.1 Rinse the Soxhlet extractors and 500 mL flat-bottom boiling flasks three times with methylene chloride. Be sure that the solvent rinses through the large vapor tube and smaller siphon tubes of the Soxhlet. Inspect these for tiny cracks. Also rinse the 24/40 lower joint.

7.1.2 Add ~ 250 mLs of methylene chloride to the 500 mL boiling flask. Add several boiling stones. Rinse the stainless steel forceps and pre-baked glass wool with Methylene chloride. Working in a hood, place a plug of the glass wool at the bottom of the Soxhlet so that the siphon tube hole is covered. Insert the 24/40 joint of the Soxhlet extractor into the 500 mL boiling flask and secure with a metal clip. Cover the top of the Soxhlet extractor with a piece of aluminum foil until ready to begin loading the sample.

7.2 Sample Handling

7.2.1 Sediment/soil samples - Decant and discard any water layer on a sediment sample. Mix the sample thoroughly with the stainless steel spatula. If the sample container is full to the extent that stirring the sample is impractical, try to remove the “best representative” aliquot from the jar based on color, particle size, moisture, etc. Discard any foreign objects such as sticks, leaves, and rocks.

7.2.2 Gummy, fibrous, or oily materials not amenable to mixing should be cut, shredded, or otherwise reduced in size to allow for maximum exposure of the sample surfaces to the extraction solvent. Materials such as glass, rubber, metal, etc. may not require mixing with powdered sodium sulfate to disperse the sample. Plastic materials must be tested for degradation (melting) in methylene chloride prior to Soxhlet extraction.

7.2.3 Please refer to Katahdin Analytical Services SOP CA-108, current revision, "Basic Laboratory Techniques" for more information of subsampling.

7.3 Weigh out a 30.00 ± 0.05 g portion of sample into a labeled 400 mL beaker. Record sample weight to nearest 0.05 g in appropriate extraction logbook. Add between 30 to 60 g of powdered sodium sulfate, as required, to produce a "free-flowing" mixture. The amount of sodium sulfate added will depend upon the moisture content of the sample (e.g., low moisture content will require less sodium sulfate). Mix well with a spatula. Keep the spatula in the sample beaker and cover the beaker with aluminum foil. Record sodium sulfate lot in logbook.

7.4 A method blank must be prepared with each extraction batch, not to exceed 20 client samples. To prepare method blank, weigh out one 30.0 ± 0.05 g portion of purified sand in a labeled 400 mL beaker. Record sample weights to nearest 0.05 g in appropriate extraction logbook. Add 60 g sodium sulfate and mix well. (Although a "free-flowing" mixture can be achieved with less than 60 g sodium sulfate, the method blank must contain 60 g in order to evaluate the sodium sulfate as a potential source of contamination.)

7.5 A laboratory control sample (LCS) must be prepared with each extraction batch, not to exceed 20 client samples. To prepare LCS, weigh out one 30.0 ± 0.05 g portion of purified sand in a labeled 400 mL beaker. Record sample weights to nearest 0.05 g in appropriate extraction logbook. Add 30 g sodium sulfate and mix well. With extraction batches prepared for combined 8081/8082 Pesticide and PCB analysis, separate Pesticide and PCB LCS's must be prepared (refer to section 5.6). If an MS/MSD pair is not extracted on a particular day, an LCS/LCSD pair may be required in order to meet client-specific or program-specific requirements. This information will be disseminated from the project manager or Department Manager.

7.6 A matrix spike/matrix spike duplicate (MS/MSD) set must be prepared for every 20 samples. To prepare MS/MSD, weigh out 30.0 ± 0.05 g portions of the sample designated for MS/MSD into each of two labeled 400 mL beakers. Record sample weights to nearest 0.05 g in appropriate extraction logbook. Add 30 g sodium sulfate to each to produce a free-flowing mixture, and mix well. With extraction batches prepared for combined 8081/8082 Pesticide and PCB analysis, separate Pesticide and PCB MS/MSD pairs must be prepared (refer to section 5.6).

7.7 Once all of the QC and field samples have been weighed and mixed with sodium sulfate, begin adding each to the assembled and appropriately labeled Soxhlet extractors using the stainless steel spatulas. Carefully scrape all of the mixtures from the beaker walls so that no more than 1% remains behind in the beaker. Be careful not to have any of the solid material fall into the extract flask through the large vapor tube.

7.8 To all samples, method blank, LCS/LCSD, and MS/MSD add 1.0 mL of the pesticide/PCBs surrogate spiking solution using a 1.0 mL gas tight syringe. Record surrogate spike volume and identification code in the extraction logbook. Thoroughly rinse syringe with solvent between each use.

7.9 To LCS/LCSD and MS/MSD add 1.0 mL of either the pesticide or PCBs matrix spike/LCS spiking solutions using a 1.0 mL gas tight syringe. Record matrix spike/LCS spiking solution volume and identification codes in the extraction logbook. Thoroughly rinse syringe with solvent between each use.

7.10 Rinse the joints of the Allihn cooling condensers with Methylene Chloride, collecting the waste in a methylene chloride solvent waste container. Place each of the Soxhlet extractors in a heating mantle and lower the Allihn cooling water condensers into the 45/50 joints of the extractors. The condensers should be set to a temperature of 15ºC. Save the pieces of aluminum foil for covering the Soxhlets when the extraction is complete. Switch on the individual heating mantles and be sure that the Rheostat of the variable transformer is set to 55% of the output voltage. Once the methylene chloride begins to boil and the Soxhlet begins to cycle (solvent will immerse the sample and collect in the Soxhlet until the level reaches that of the small siphon tube and then begin to spill over into the extract flask), re-check the apparatus' for leaks. Allow the samples to extract for 18-24 hours. Be sure the chiller/recirculator temperature is set low enough to provide enough cooling capacity for the number of extractions in the batch.

7.11 When the extraction is complete, allow the extracts to cool before dismantling. Remove the Allihn condenser and replace the aluminum foil on top of the extractor. Move the extractors to a hood and detach the extractor from the extract flask. Tilt each extractor slightly to cause any remaining solvent in the sample chamber to drain through the siphon tube into the extract flask. This will help to cool the extract flask and make the apparatus easier to dismantle. Try to drain as much solvent as possible from the extractor into the flask. **This is done by rinsing a glass tube in methylene chloride and pressing on the sample slightly so that as solvent as possible is drained into the extract flask.** Cover the flask with aluminum foil and store in the interim extract storage refrigerator unless the extracts are to be concentrated the same day.

7.12 Immediately remove the extracted soil/sodium sulfate mixtures from the extractors using a square edge spatula, and dispose of in an appropriate solid waste container. It is important to do this soon after the extractors are dismantled, as the sample mixture will tend to "freeze" into a solid mass in the Soxhlet as the solvent dries.

CONCENTRATION OF THE EXTRACTS

7.13 Rinse the K-D glassware (flask, concentration tube, and Snyder column, including the ground glass joints on the flask and columns) three times with methylene chloride before assembling. Add a few boiling chips to the K-D. Insert fluted 18.5 cm filter papers into short stem powder funnels and add ~ 2 inches of sodium sulfate crystals. Rinse the sodium sulfate in the assembled funnels with ~20-30 mLs of methylene chloride. Place the assembled K-D's under the funnels. Record the lot numbers of the solvent, sodium sulfate and filter papers in the extraction logbook.

7.14 If samples are to be GPC'd, refer to the current revision of Katahdin SOP CA-513, Extract Cleanup Using Gel Permeation Chromatography, for appropriate concentrating procedures. Samples that undergo GPC are not solvent exchanged into hexane.

7.15 If samples are not to be GPC'd follow Steps 7.16 through 7.23 to concentrate extracts to final volume of 10.0 mLs

7.16 For a solvent exchange, (for samples not being GPC'd), add approximately 50 mL hexane to funnel and let drain through. Since methylene chloride has a lower boiling point than Hexane, this will result in a final extract in hexane only. Record the lot number of the solvent in the extraction logbook.

7.17 Transfer the extracts to the K-D concentrator setups through the sodium sulfate in the funnels. This is the drying step, which is required to remove residual water from the extracts. Any large water layers must be removed by other means, prior to pouring through the sodium sulfate. After pouring all of the extract volume through the sodium sulfate, rinse the extract flask three times with ~ 2 – 3 mLs of methylene chloride. Add the rinsings through the sodium sulfate to complete a quantitative transfer. Rinse the sodium sulfate with ~ 15 mLs of methylene chloride and allow to drain.

7.18 Transfer the labels from the extract flasks to the K-Ds. Remove the funnel and attach a 3-ball macro Snyder column. Pre-wet the Snyder column with 1 mL of methylene chloride.

7.19 Place the K-D in a hot water bath (75-85°C). Gently swirl K-D in the water until boiling begins. At the proper distillation rate, the Snyder balls should chatter but the chambers should not flood with condensed solvent. The K-D should be kept in a vertical orientation while on the bath. When the apparent volume in the concentrator tube reaches ≈ 6 mL, remove the K-D from the water bath. Allow the K-D to cool for 10 minutes. Rinse the Snyder column lower joint with ≈ 1 mL of methylene chloride, hexane, if exchange is taking place. Remove the Snyder column. Wipe off any water from the neck above the lower joint of the flask.

Separate the K-D flask from the concentrator tube, rinsing the ground glass joint with ≈ 1 mL methylene chloride, hexane, if exchange is taking place.

7.20 Reduce the extract in the concentrator tube to approximately 1-2 mL using the nitrogen blow-down apparatus to ensure all methylene chloride has been evaporated. The bath temperature must be no higher than the boiling point of the solvent (39°C for methylene chloride). Turn the gas to 3 psi. Be careful not to splash the extract out of the tube. During concentration on the N-evap, the internal wall of the concentrator tube and the N-evap sparging pipet must be rinsed down at least once or twice with ≈1 mL of methylene chloride. The solvent level in the concentrator tube must be positioned below the level of the water bath as much as possible to prevent water from condensing into the sample extract. As the extract volume is reduced, lower the N_2 sparging pipet closer to the surface of the extract to expedite the concentration. Record the temperature of the water in the nitrogen evaporation water bath in the extraction logbook also note any problems or extract losses, if they occur..

7.21 Complete quantitative transfer of the extract to a vial by using hexane. Adjust the volume of the hexane extract to 10 mL in either a 12 or 16 mL vial using the appropriate “reference vial” for volume comparison.

7.22 Label the vial with lab sample number, extraction date, matrix and analysis. Store extract vials at a temperature of 4 ± 2 °C until ready for analysis. Indicate in the extractions logbook the box number and “tray location” of the individual extract vials.

7.23 All sample extracts for 8082 PCB analysis must undergo a sulfuric acid wash (cleanup) prior to analysis, unless it has been GPC’d. All sample extracts for 8081 pesticide analysis should undergo further cleanup using the GPC unless time is a factor. All sample extracts for combined 8081/8082 analyses must be split unless GPC’d. One portion must be acid cleaned for 8082 analysis. The associated method blank must be split and acid-cleaned in the same fashion. PCB LCSs and matrix spikes are acid cleaned also. Pesticide LCSs and matrix spikes are not subjected to further cleanup. Record the lot number of the acid in the extraction logbook. Please refer to Katahdin SOP CA525 (current revision), Extract Cleanup Using Sulfuric Acid, for further instructions.

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

A method blank must be extracted for each and every item listed below:

- Each sample matrix (soil, water)
- Each day of extraction (24 hours midnight - midnight)
- Each extraction method or level
- Every 20 samples extracted in a 24-hour period

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SOXHLET EXTRACTION USING METHOD 3540 FOR PESTICIDE/PCB ANALYSIS**

A laboratory control sample (LCS) is required for each and every item listed below:

- Each sample matrix
- Each extraction method or level
- Every extraction batch of twenty or fewer samples
- Each analysis (pesticide and/or PCB) to be performed

Refer to the current revision of the applicable Katahdin SOP for analysis of Pesticides and PCBs for quality control acceptance criteria.

Each extractions analyst must demonstrate proficiency in performing the extractions that prepare samples for analysis. Demonstration consists of preparation (extraction), by the analyst, of at least four aliquots which are then analyzed according to the analytical method in question. These QC samples must meet all quality control acceptance limits. Demonstration must be documented by use of a form which summarizes the results of the analysis of these aliquots, calculated percent recoveries, and standard deviation. Demonstration of proficiency must be done one time per analyst initially and then annually thereafter. Refer to SOPs QA-805 and QA-807, current revision.

If, upon analysis of the extracted samples, it is discovered that quality control acceptance criteria have not been met, all associated samples must be evaluated against all the QC. In some cases data may be reported, perhaps with narration, while in other cases, other corrective action may be taken. The corrective actions may include re-extraction of the samples associated with the quality control sample that did not meet acceptance criteria, or may include making new reagents and standards if the standardization is suspect. These decisions are based on holding time considerations, client and project specific Data Quality Objectives and on review of chromatograms. The supervisor, Operations Manager, and/or Quality Assurance Officer may be consulted to evaluate data. Some samples may not be able to be reanalyzed within hold time. In these cases "qualified" data with narration may be advisable after consultation with the client.

Much of the work performed at the lab is analyzed in accordance with specific QC requirements spelled out in a project specific Quality Assurance Project Plan (QAPP) or in a program specific Quality Systems Manual (QSM). The reporting limits, acceptance criteria and/or corrective actions may be different than those specified in this SOP. In these cases the appropriate information will be communicated to the Department Manager and/or senior chemists before initiation of the analyses so that specific product codes can be produced for the project. In addition, the work order notes for each project will describe the specific QAPP or QSM to be followed.

9.0 METHOD PERFORMANCE

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SOXHLET EXTRACTION USING METHOD 3540 FOR PESTICIDE/PCB ANALYSIS**

The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero. The MDLs are determined annually per type of instrument and filed with the Organic Department Manager and with the QAO. Refer to the current revision of Katahdin SOP QA-806, Method Detection Limit, Method Detection Limit, Instrument Detection Limit and reporting Limit Studies and Verifications, for procedures on determining the MDL.

Refer to the applicable analytical SOP for other method performance parameters and requirements.

10.0 APPLICABLE DOCUMENTS/REFERENCES

Test Methods for Evaluating Solid Waste-Physical/Chemical Methods, Method 3540C, SW-846, Third Edition, Updates I, II, IIA, IIB, and III Revised December 1996, US EPA.

Department of Defense Quality Systems Manual for Environmental Laboratories (DOD QSM), Version 4.1, 04/22/09.

The National Environmental Laboratory Accreditation Conference (NELAC) Standards, June 2003.

LIST OF TABLES AND FIGURES

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SOXHLET EXTRACTION USING METHOD 3540 FOR PESTICIDE/PCB ANALYSIS**

TABLE 1

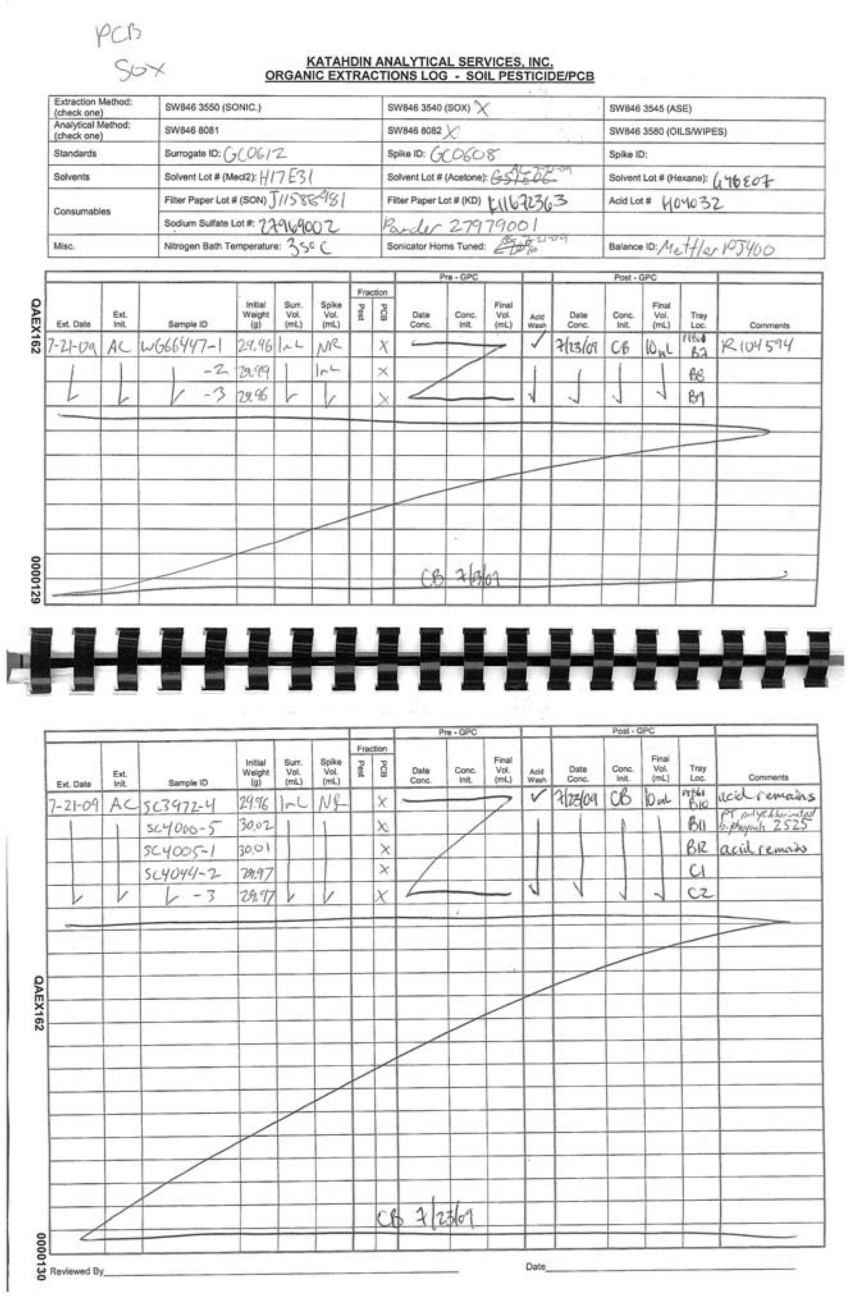
SUMMARY OF METHOD MODIFICATIONS

TOPIC	KATAHDIN SOP CA-524-06	METHOD 3540, current revision
Apparatus/Materials	1. short stem funnels	1. drying columns
Reagents		
Sample preservation/ handling		
Procedures	1. Use 30 grams of sample and 30 grams of sodium sulfate. 2. Use 250 mL of methylene chloride 3. no apparatus height specification for concentration on water bath 4. water bath at 75-85 deg C 5. sample removed from water bath when volume reaches ~6 mL 6. Solvent exchange to hexane is performed using K-D apparatus with addition of approximately 50 mL hexane at the start of concentration process	1. Use 10 grams of sample and 10 grams of sodium sulfate. 2. Use 300 mL of methylene chloride 3. partially immerse concentrator tube in water and lower apparatus to complete concentration in 10-15min 4. water bath at 80-90 deg C 5. sample removed from water bath when volume reaches 1-2 mL 6. Solvent exchange to hexane is performed using K-D apparatus with addition of approximately 50 mL hexane after concentrating methylene chloride extract to 1 mL
QC - Spikes		
QC - LCS		
QC - Accuracy/Precision		
QC - MDL		

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SOXHLET EXTRACTION USING METHOD 3540 FOR PESTICIDE/PCB ANALYSIS**

FIGURE 1

EXAMPLE OF LOGBOOK PAGE



PCB
SOX

KATAHDIN ANALYTICAL SERVICES, INC.
ORGANIC EXTRACTIONS LOG - SOIL PESTICIDE/PCB

Extraction Method: (check one)	SW846 3550 (SONIC.)	SW846 3540 (SOX) X	SW846 3545 (ASE)
Analytical Method: (check one)	SW846 8081	SW846 8082 X	SW846 3580 (OILS/WIPES)
Standards	Surrogate ID: GC0612	Spike ID: GC0608	Spike ID:
Solvents	Solvent Lot # (Mecl2): H17E31	Solvent Lot # (Acetone): G51E06	Solvent Lot # (Hexane): G46E07
Consumables	Filter Paper Lot # (SON) J1158981	Filter Paper Lot # (KD) K1162363	Acid Lot # H04032
	Sodium Sulfate Lot #: 27969002	Powder 27979001	
Misc.	Nitrogen Bath Temperature: 35° C	Sonicator Horns Tuned:	Balance ID: Mettler PJ400

QAEX162 0000129

Ext. Date	Ext. Init.	Sample ID	Initial Weight (g)	Surr. Vol. (mL)	Spike Vol. (mL)	Fraction Pest	Fraction PCB	Pre-GPC Date Conc.	Pre-GPC Conc. Init.	Pre-GPC Final Vol. (mL)	Acid Wash	Post-GPC Date Conc.	Post-GPC Conc. Init.	Post-GPC Final Vol. (mL)	Tray Loc.	Comments
7-21-09	AC	WG66447-1	29.96	1mL	NR		X				✓	7/23/09	CB	10mL	B2	R104594
↓	↓	-2	29.99		1mL		X				↓	↓	↓	↓	B8	
↓	↓	-3	29.96	↓	↓		X				↓	↓	↓	↓	B9	

CB 7/23/09

QAEX162 0000130

Ext. Date	Ext. Init.	Sample ID	Initial Weight (g)	Surr. Vol. (mL)	Spike Vol. (mL)	Fraction Pest	Fraction PCB	Pre-GPC Date Conc.	Pre-GPC Conc. Init.	Pre-GPC Final Vol. (mL)	Acid Wash	Post-GPC Date Conc.	Post-GPC Conc. Init.	Post-GPC Final Vol. (mL)	Tray Loc.	Comments
7-21-09	AC	SC3972-4	29.96	1mL	NR		X				✓	7/23/09	CB	10mL	B10	acid remains
↓	↓	SC4000-5	30.02	↓	↓		X				↓	↓	↓	↓	B11	PT polychlorinated biphenyls 2525
↓	↓	SC4005-1	30.01	↓	↓		X				↓	↓	↓	↓	B12	acid remains
↓	↓	SC4044-2	29.97	↓	↓		X				↓	↓	↓	↓	C1	
↓	↓	-3	29.97	↓	↓		X				↓	↓	↓	↓	C2	

CB 7/23/09

Reviewed By__________ Date__________

TITLE: PREPARATION OF SEDIMENT/SOIL SAMPLES BY SOXHLET EXTRACTION USING METHOD 3540 FOR SUBSEQUENT EXTRACTABLE SEMIVOLATILE ANALYSIS

Prepared By: Mike Thomas Date: 7/98

Approved By:

Group Supervisor: Michael Thomas Date: 11/15/00

Operations Manager: [signature] Date: 11/15/00

QA Officer: Deborah J. Nadeau Date: 11·16·00

General Manager: [signature] Date: 11/20/00

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
01	Minor changes throughout. Clarifications to procedure section.	DN	11.16.00	11/16/00
02	Definitions added to section 1.1. Wording was added or changed to clarify sections 4, 5, 6, 7, 8 & 9. Minor changes throughout. New figures.	MRC	11.09.04	11.09.04
03 LAD 6-26-06	Updated Sect. 7.0 to include SIM. Updated figures 2 and 3 to include current SVOA compounds ~~MIXES~~ used. Updated Sect. 5.0 to include all compounds analyzed for. Updated logbook page. minor edits throughout.	LAD	04/06	04/06
04	Added waste generated information. Updated Spikes and Surrogates. Added SIM LCSD and MS/D requirements. Updated Table 1. Added GPC references. Added LCSD after LCS.	LAD	09/07	09/07
05	Updated logbook page. Added acidpate compounds to Fig. 2. Added recording of consomable's lot #'s and recording the Nitrogen water bath temp. in logbook	LAD	07/08	07/08

TITLE: PREPARATION OF SEDIMENT/SOIL SAMPLES BY SOXHLET EXTRACTION USING METHOD 3540 FOR SUBSEQUENT EXTRACTABLE SEMIVOLATILE ANALYSIS

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
06	Added balance criteria. Changed all weight criteria to +/- 0.05g. Revised section 7 to reflect current techniques. Added SOP CA-108 reference for sub-sampling. Updated logbook example	LAD	08/09	08/09

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SOXHLET EXTRACTION USING METHOD 3540 FOR SUBSEQUENT EXTRACTABLE SEMIVOLATILE ANALYSIS**

I acknowledge receipt of copy ___ of document **SOP CA-526-06**, titled **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SOXHLET EXTRACTION USING METHOD 3540 FOR SUBSEQUENT EXTRACTABLE SEMIVOLATILE ANALYSIS.**

Recipient: _____ Date: _____

KATAHDIN ANALYTICAL SERVICES, INC.
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy ___ of document **SOP CA-526-06**, titled **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SOXHLET EXTRACTION USING METHOD 3540 FOR SUBSEQUENT EXTRACTABLE SEMIVOLATILE ANALYSIS.**

Recipient: _____ Date: _____

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SOXHLET EXTRACTION USING METHOD 3540 FOR SUBSEQUENT EXTRACTABLE SEMIVOLATILE ANALYSIS**

1.0 SCOPE AND APPLICATION

The purpose of this SOP is to describe the procedures and requirements f or extracting semivolatile organic compounds from solids such as soils, sludges, and wastes using Method 3540. The Soxhlet extraction process ensures intimate contact the sample matrix with the extraction solvent.

This method is applicable to the isolation and concentration of water-insoluble and slightly water soluble organics in preparation for a variety of chromatographic procedures.

1.1 Definitions

METHOD BLANK (laboratory reagent blank): An artificial sample designed to determine if method analytes or other interferences are present in the laboratory environment, the reagents, or the apparatus. For aqueous samples, reagent water is used as a blank matrix; for soil samples, baked organic-free sand is used as a blank matrix. The blank is taken through the appropriate steps of the process.

LABORATORY CONTROL SAMPLE (LCS): A blank that has been spiked with the analyte(s) from an independent source, and is analyzed exactly like a sample. Its purpose is to determine whether the methodology is in control, and whether the laboratory is capable of making accurate and precise measurements. The matrix used should be phase matched with the samples and well characterized.

MATRIX SPIKE/MATRIX SPIKE DUPLICATE (MS/MSD): Predetermined quantities of stock solutions of certain analytes are added to a sample matrix prior to sample extraction and analysis. Samples are split into duplicates, spiked and analyzed. Percent recoveries are calculated for each of the analytes detected. The relative percent difference between the samples is calculated and used to assess analytical precision.

SURROGATES: Organic compounds which are similar to analytes of interest in chemical composition, extraction and chromatography, but which are not normally found in environmental samples. These compounds are spiked into all blanks, standards, samples and spiked samples prior to analysis. Percent recoveries are calculated for each surrogate.

1.2 Responsibilities

This method is restricted to use by, or under the supervision of analysts experienced in the extraction of samples for semivolatile analysis. Each analyst must demonstrate and document their ability to generate acceptable results with this method. Refer to Katahdin SOP QA-805, current revision, “Personnel Training and Documentation of Capability”.

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SOXHLET EXTRACTION USING METHOD 3540 FOR SUBSEQUENT EXTRACTABLE SEMIVOLATILE ANALYSIS**

It is the responsibility of all Katahdin technical personnel involved in the extraction of samples for semivolatile analysis to read and understand this SOP, adhere to the procedures outlined, and to properly document their data in the appropriate lab notebook. Any deviations from the test or irregularities with the samples should also be recorded in the lab notebook and reported to the Department Manager or designated qualified data reviewer responsible for this data.

It is the responsibility of the Department Manager to oversee that the members of his/her group follow this SOP, to assure that their work is properly documented, and to indicate periodic review of the pertinent logbooks.

1.3 Safety

Users of this procedure must be cognizant of inherent laboratory hazards, proper disposal procedures for contaminated materials and appropriate segregation of hazardous wastes. The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical should be treated as a potential health hazard. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Everyone involved with the procedure must be familiar with the MSDSs for all the materials used in this procedure.

Each qualified analyst or technician must be familiar with Katahdin Analytical Environmental Health and Safety Manual including the Katahdin Hazardous Waste Plan and must follow appropriate procedures. These include the use of appropriate personal protective equipment (PPE) such as safety glasses, gloves and lab coats when working with chemicals or near an instrument and not taking food or drink into the laboratory. Each analyst should know the location of all safety equipment. Each analyst shall receive a safety orientation from their Department Manager, or designee, appropriate for the job functions they will perform.

1.4 Waste Disposal

Wastes generated during the preparation of samples must be disposed of in accordance with the procedures described in the current revision of the Katahdin Hazardous Waste Plan and Safety Manual and SOP SD-903, “Sample Disposal,” current revision. Expired standards are lab packed, placed in the Katahdin hazardous waste storage area, and disposed of in accordance with this SOP.

Any methylene chloride solvent waste generated during the rinsing of glassware etc. should be disposed of in the “D” waste stream satellite accumulation area nearest the point of generation. Acetone and methanol are considered flammable waste, and should be disposed of in the “O” waste stream satellite accumulation area nearest the point of generation. Post-extraction soil samples, used glass wool, and sodium sulfate waste should be disposed of in the soil with organics “I” waste stream satellite

accumulation area nearest the point of generation. Please refer to the current revision of SOP CA-107 for the location of satellite waste accumulation areas.

2.0 SUMMARY OF METHOD

2.1 The solid sample is mixed with anhydrous sodium sulfate, placed in a Soxhlet extractor and extracted with methylene chloride.

2.2 The extract is then dried and concentrated for subsequent 8270 Semivolatile Organics analysis.

3.0 INTERFERENCES

Contaminants in solvents, reagents, glassware, and other sample processing hardware may cause method interferences such as discrete artifacts and/or elevated baselines in the total ion current profiles (TICPs). All of these materials routinely must be demonstrated to be free from interferences under the conditions of the analysis by running laboratory method blanks. Interferences caused by phthalate esters can pose a major problem in semivolatile organics analysis because many phthalates are also target analytes. Common flexible plastics contain varying amounts of phthalates that are easily extracted during laboratory operations, so cross-contamination of glassware frequently occurs when plastics are handled. Interferences from phthalates can best be minimized by avoiding the use of such plastics in the laboratory. At no time may gloves that have not been tested for phthalates or gloves known to contain phthalates be used or stored in the organic extraction lab. Additionally, whenever possible plastic items in this lab must be replaced with metal or Teflon or other non-phthalate plastic substitute.

Special care should be taken to ensure clean glassware and apparatus are used, pre-rinsed with the appropriate solvent prior to use. Solvents should be analyzed prior to use to demonstrate that each lot is free of contaminants that may interfere with the analysis.

Interferences coextracted from the samples will vary considerably from source to source. If analysis of an extracted sample is prevented due to interferences, further cleanup of the sample extract may be needed to minimize interferences.

4.0 APPARATUS AND MATERIALS

4.1 Soxhlet apparatus:

a) Soxhlet extractor – 45/50 top joint and 24/40 lower joint.
b) 500 mL flat-bottom boiling flask

c) Allihn cooling water condenser

4.2 Powder Funnels – 100 mm top diameter, 35 mm stem

4.3 Kuderna-Danish (K-D) apparatus

4.3.1 Concentrator tube - 10-mL

4.3.2 Evaporation flask - 500-mL

4.3.3 Snyder column - Three-ball macro

4.4 Nitrogen evaporation (N-EVAP) apparatus.

4.5 Boiling stones, 12 mesh silicon carbide (carborundum) – pre-purified by Soxhlet extraction in methylene chloride

4.6 Water bath - Heated, with concentric ring cover, capable of temperature control (± 5°C). The bath should be used in a hood.

4.7 Vials - Glass, 1.8-mL capacity, with polytetrafluoroethylene (PTFE)-lined septum vials, and 12 mL with Teflon-lined caps for extracts designated for GPC cleanup.

4.8 Glass wool (fiberglass) - baked at 400°C for a minimum of 4 hours or overnight.

4.9 Heating mantles - Rheostat controlled.

4.10 Disposable glass pasteur pipets, 5 ¾" and bulbs.

4.11 Drying oven - capable of maintaining 105°C for glassware drying.

4.12 Muffle oven – capable of maintaining 400 °C for baking glass wool and organic-free sand.

4.13 Beakers, 250 or 400 mL

4.14 Top-loading balance - capable of weighing to 0.**01** g.

4.15 Spatulas, stainless-steel

4.16 Long forceps, stainless-steel

4.17 Metal clips – for securing Soxhlets to boiling flasks

4.18 Filter Paper, 18.5 cm, Fisherbrand or Whatman 114V (or equivalent)

5.0 REAGENTS

5.1 Sodium Sulfate - anhydrous powdered and granular crystals, reagent grade, certified by the manufacturer/vendor as purified heating to 400°C prior to receipt by the laboratory.

5.2 Methylene chloride, methanol, and acetone - pesticide residue analysis grade or equivalent. Methylene chloride and acetone are evaluated, by lot, prior to use, by concentration of approximately 400 mL to 1.0 mL followed by GC/MS analysis.

5.3 Organic-free sand, purified by baking at 400 °C. Method blanks serve as checks on the baked sand.

5.4 Base/Neutral and Acid (SVOA) Surrogate Spiking Solution - Surrogate standards are added to all samples and calibration solutions. Prepare a surrogate standard spiking solution that contains the following compounds at the indicated concentrations in acetone.

Compound	Conc.
phenol-$_{d6}$	100 ug/mL
2,4,6-tribromophenol	100 ug/mL
2-fluorophenol	100 ug/mL
nitrobenzene-$_{d5}$	50 ug/mL
p-terphenyl-$_{d14}$	50 ug/mL
2-fluorobiphenyl	50 ug/mL

Store the spiking solution at -10°C to -20°C in Teflon-sealed containers. These solutions must be replaced after six months, or sooner if comparison with quality control check samples indicates a problem. If reanalysis of a method blank still indicates surrogates out of criteria, a new surrogate solution must be used.

5.5 SIM Surrogate Spiking Solution- Surrogate Standards are added to all samples and calibration solutions. Prepare a surrogate solution that contains the following compounds at a concentration of 2 ug/mL in acetone.

Compound	Conc. ug/mL
Fluorene-d10	2.0 ug/mL
2-Methylnaphthalene-d10	2.0 ug/mL
Pyrene-d10.	2.0 ug/mL
2,4-Dibromophenol	2.0 ug/mL

Store the spiking solution at -10°C to -20°C in Teflon-sealed containers. These

solutions must be replaced after six months, or sooner if comparison with quality control check samples indicates a problem. If reanalysis of a method blank still indicates surrogates out of criteria, a new surrogate solution must be used.

5.6 Base/Neutral and Acid (SVOA) Lab Control Sample / Matrix Spike Spiking Solution - Prepare a spiking solution in methanol that contains the following mixes listed in Figure 2 at a concentration of 50 ug/ml for the base/neutral compounds and 100 ug/ml for the acid compounds. Store the spiking solution at -10°C to -20°C in Teflon-sealed containers. These solutions must be replaced after six months, or sooner if comparison with quality control check samples indicates a problem.

5.7 Base/Neutral (SIM) Matrix Spike/ Lab Control Sample Spike Solution for SIM-SVOA. Prepare a spiking solution in methanol that contains the compounds listed in Figure 2 at a concentration of 2 ug/mL for base/neutral. Take out 1.0 mL of Base/Neutral and Acid Matrix Spike/Lab Control Spiking Solution for SVOA and dilute it to 25.0 mL in methanol. Store the solution Spiking at -10°C to -20°C in Teflon-sealed containers. These solutions must be replaced after six months, or sooner if comparison with quality control check samples indicates a problem.

5.8 Base/Neutral and Acid (SVOA) Appendix IX Lab Control Sample / Matrix Spike Spiking Solution – Prepare a spiking solution in methanol that contains the compounds listed in Figure 3 at concentrations of 100 ug/ml. Store the spiking solution at -10°C to -20°C in Teflon-sealed containers. These solutions must be replaced after six months, or sooner if comparison with quality control check samples indicates a problem.

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

Sediment/soil samples must be collected in a soil jar and must be maintained at 4°C (±2°C).

Holding time for extraction of sediment/soil samples for Method 3540 is 14 days from date of sample collection, although the analyst should be aware that actual holding times employed may be project/program specific.

Store all extracts at 4°C (±2°C) in the dark in labeled Teflon-sealed containers. See SOP SD-902, "Sample Receipt and Internal Control," current revision, for storage areas and temperature maintenance procedures.

7.0 PROCEDURES

All solid samples need to be cleaned up to reduce matrix interferences, time permitting. The cleanup procedure employed is gel permeation chromatography (GPC).

Sign chain-of-custody when removing and replacing samples in storage locations, and fill out the sample preparation/extraction log with the necessary information before starting the extraction. Prerinse all glassware three times with methylene chloride.

7.1 Preparing the Soxhlet Extraction Apparatus

7.1.1 Rinse the Soxhlet extractors and 500 mL flat-bottom boiling flasks three times with methylene chloride. Be sure that the solvent rinses through the large vapor tube and smaller siphon tubes of the Soxhlet. Inspect these for tiny cracks. Also rinse the 24/40 lower joint.

7.1.2 Add ~ 250 mLs of methylene chloride to the 500 mL boiling flask. Add several boiling stones. Using stainless steel forceps and working in a hood, place a plug of the pre-baked glass wool at the bottom of the Soxhlet so that the siphon tube hole is covered. Insert the 24/40 joint of the Soxhlet extractor into the 500 mL boiling flask and secure with a metal clip. Cover the top of the Soxhlet extractor with a piece of aluminum foil until ready to begin loading the sample. Record the solvent lot number in the extraction logbook.

7.2 Sample Handling

7.2.1 Sediment/soil samples - Decant and discard any water layer on a sediment sample. Mix the sample thoroughly with the stainless steel spatula. If the sample container is full to the extent that stirring the sample is impractical, try to remove the “best representative” aliquot from the jar based on color, particle size, moisture, etc. Discard any foreign objects such as sticks, leaves, and rocks.

7.2.2 Gummy, fibrous, or oily materials not amenable to mixing should be cut, shredded, or otherwise reduced in size to allow for maximum exposure of the sample surfaces to the extraction solvent. Materials such as glass, rubber, metal, etc. may not require mixing with powdered sodium sulfate to disperse the sample. Plastic materials must be tested for degradation (melting) in methylene chloride prior to Soxhlet extraction.

7.2.3 Refer to Katahdin SOP CA-108, current revision, “Basic Laboratory Technique” for more information on subsampling.

7.3 The following steps should be performed rapidly to avoid loss of the more volatile extractables. Weigh out a 30.00 ± 0.05 g portion of sample into a labeled 400-mL beaker. Record sample weight to the nearest 0.05 g in appropriate extraction logbook. Add between 30 g and 60 g of anhydrous powdered sodium sulfate as

required to produce a "free-flowing" mixture. The amount of sodium sulfate added will depend upon the moisture content of the sample (e.g., low moisture content will require less sodium sulfate). Mix well with a spatula. Keep the spatula in the sample beaker and cover the beaker with aluminum foil.

7.4 A method blank must be prepared with each extraction batch, not to exceed 20 client samples. To prepare method blank, weigh out one 30.00 ± 0.05 g portion of purified sand in a labeled 400 mL beaker. Add 60 g sodium sulfate and mix well. Although a "free-flowing" mixture can be achieved with less than 60 g sodium sulfate, the method blank must contain 60 g in order to evaluate the sodium sulfate as a potential source of contamination.

7.5 A laboratory control sample (LCS) must be prepared with each extraction batch, not to exceed 20 client samples. To prepare LCS, weigh out one 30.00 ± 0.05 g portion of purified sand in a labeled 400 mL beaker. Add 30 g sodium sulfate and mix well. If combined SIM-SVOA analysis is requested, a separate LCS must be prepared for each analysis. If an MS/MSD pair is not extracted on a particular day, an LCS/LCSD pair may be required in order to meet client-specific or program-specific requirements. This information will be disseminated from the project manager or Department Manager.

7.6 A matrix spike/matrix spike duplicate (MS/MSD) set must be prepared for every 20 samples. To prepare MS/MSD, weigh out 30.00 ± 0.05 g portions of the sample designated for MS/MSD into each of two labeled 400 mL beakers. Record sample weights to nearest 0.05 g in appropriate extraction logbook. Add 30 - 60 g sodium sulfate to each to produce a free-flowing mixture, and mix well. If combined SIM-SVOA analysis is requested, a separate MS/MSD must be prepared for each analysis.

7.7 Once all of the QC and field samples have been weighed and mixed with sodium sulfate, begin adding each to the assembled and appropriately labeled Soxhlet extractors using the stainless steel spatulas. Carefully scrape all of the mixtures from the beaker walls so that no more than 1% remains behind in the beaker. Be careful not to have any of the solid material fall into the extract flask through the large vapor tube.

7.8 To all samples, method blank, LCS/LCSD, and MS/MSD add 1.0 mL of the appropriate base/neutral and acid surrogate spiking solution listed below using the pre-rinsed 1.0 mL gas tight syringe. Record surrogate spike volume and identification code in extraction logbook. Thoroughly rinse syringe with solvent prior to using it for another spiking solution.

7.8.1 If the request is for SVOA, use the SVOA surrogate solution (sect. 5.4)

7.8.2 If the request is for SIM, use the SIM surrogate solution (sect. 5.5).

7.8.3 If the request is for SIM-SVOA, use both the SIM and SVOA surrogate solutions. NOTE: Separate LCS/LCSD and/or MS/MSD are needed for each analysis and should be spiked with the appropriate surrogate.

7.9 To the LCS/LCSD and the MS/MSD add 1.0 mL of the appropriate base/neutral and acid (SVOA) matrix spike/LCS spiking solution listed below using a 1.0 mL gas tight syringe. Record matrix spike/LCS spiking solution volume and identification code in extraction logbook. Thoroughly rinse syringe with solvent when spiking is completed.

7.9.1 If the request is for SVOA, add 1 mL of the SVOA spiking solution (sect. 5.6).

7.9.2 If the request is for SIM, add 1 mL of the SIM Spiking solution (sect. 5.7).

7.9.3 If the request is for SVOA/SIM, add 1 mL of the SVOA spiking solution and 1 mL of the SIM Spiking solution to appropriate LCS/LCSD and/or MS/MSD. (sect's 5.6 and 5.7).

7.9.4 If the request is for SVOA Appendix IX , add 1 mL of the SVOA Appendix IX spiking solution and 1 mL of the SVOA spiking solution (sect's 5.6 and 5.8).

7.10 Place each of the Soxhlet extractors in a heating mantle and lower the Allihn cooling water condensers into the 45/50 joints of the extractors. Save the pieces of aluminum foil for covering the Soxhlets when the extraction is complete. Switch on the individual heating mantles and be sure that the Rheostat of the variable transformer is set to 55-60% of the output voltage. Once the methylene chloride begins to boil and the Soxhlet begins to cycle (solvent will immerse the sample and collect in the Soxhlet until the level reaches that of the small siphon tube and then begin to spill over into the extract flask), re-check the apparatus' for leaks. Allow the samples to extract for 18-24 hours. Be sure the chiller/recirculator temperature is set low enough to provide enough cooling capacity for the number of extractions in the batch.

7.11 When the extraction is complete, allow the extracts to cool before dismantling. Tilt each extractor slightly to cause any remaining solvent in the sample chamber to drain through the siphon tube into the extract flask. This will help to cool the extract flask and make the apparatus easier to dismantle. Remove the Allihn condenser and replace the aluminum foil on top of the extractor. Move the extractors to a hood and detach the extractor from the extract flask. Try to drain as much solvent as possible from the extractor into the flask. **This is done by rinsing a glass tube in methylene chloride and pressing on the sample slightly so that as solvent as possible is drained into the extract flask.** Cover the flask with aluminum foil and

store in the interim extract storage refrigerator unless the extracts are to be concentrated the same day.

7.12 Immediately remove the extracted soil/sodium sulfate mixtures from the extractors using a square edge spatula, and dispose of in an appropriate solid waste container. It is important to do this soon after the extractors are dismantled, as the sample mixture will tend to "freeze" into a solid mass in the Soxhlet as the solvent dries.

CONCENTRATION OF EXTRACTS

7.13 Rinse the K-D glassware (flask, concentration tube, and Snyder column, including the ground glass joints on the flask and columns) three times with methylene chloride before assembling. Add two boiling chips to the K-D. Insert 18.5 cm filter papers into short stem powder funnels and add ~ 2 inches of sodium sulfate crystals. Rinse the sodium sulfate in the assembled funnels with ~20-30 mLs of methylene chloride. Place the assembled K-D's under the funnels. Record the filter paper and sodium sulfate lot numbers in the extraction logbook.

7.14 Transfer the extracts to the K-D concentrator setups through the sodium sulfate in the funnels. This is the drying step, which is required to remove residual water from the extracts. Any large water layers must be removed by other means, prior to pouring through the sodium sulfate. After pouring all of the extract volume through the sodium sulfate, rinse the extract flask three times with ~ 2 – 3 mLs of methylene chloride. Add the rinsings through the sodium sulfate to complete a quantitative transfer. Rinse the sodium sulfate with ~ 15 mLs of methylene chloride and allow draining.

7.15 All samples should go through GPC cleanup except if time does not permit. Refer to the current revision of Katahdin SOP CA-513, Extract Cleanup Using Gel Permeation Chromatography, for appropriate concentrating procedures.

7.16 If samples are not to be GPC'd, when time does not permit, follow Steps 7.17 through 7.22 to concentrate extracts to final volume of 1 mL.

7.17 Transfer the labels from the extract flasks to the K-Ds. Remove the funnel and attach a 3-ball macro Snyder column. Pre-wet the Snyder column with 1 mL of methylene chloride.

7.18 Place the K-D in a hot water bath (75-85°C). Gently swirl K-D in the water until boiling begins. At the proper distillation rate, the Snyder balls should chatter but the chambers should not flood with condensed solvent. The K-D should be kept in a vertical orientation while on the bath. When the apparent volume in the concentrator tube reaches ≈ 4-6 mL, remove the K-D from the water bath. **Do not allow the evaporator to go dry. If the sample extract does go dry, re-**

extraction must occur immediately. Allow the K-D to cool for 10 minutes. Rinse the Snyder column lower joint with ≈ 1 mL of methylene chloride. Remove the Snyder column. Wipe off any water from the neck above the lower joint of the flask. Separate the K-D flask from the concentrator tube, rinsing the ground glass joint with ≈ 1 mL methylene chloride.

7.19 Reduce the extract in the concentrator tube to approximately 1 mL using the nitrogen blow-down apparatus. The bath temperature must be no higher than the boiling point of the solvent (39 °C for methylene chloride). Turn the gas to 3 psi. Be careful not to splash the extract out of the tube. During concentration on the N-evap, the internal wall of the concentrator tube and the N-evap sparging pipet must be rinsed down at least once or twice with ≈1 mL of methylene chloride. The solvent level in the concentrator tube must be positioned below the level of the water bath as much as possible to prevent water from condensing into the sample extract. As the extract volume is reduced, lower the N_2 sparging pipet closer to the surface of the extract to expedite the concentration. Record the temperature of the water in the nitrogen evaporation water bath in the extraction logbook, also note any problems or extract losses, if they occur.

7.20 When the apparent volume reaches slightly less than 1 mL, remove the concentrator tube and allow it to cool.

7.21 Complete the quantitative transfer of the extract to a 1.8 mL vial by using methylene chloride. Adjust the volume of the methylene chloride extract to 1.0 mL using the 1.8 mL reference vial for volume comparison.

7.22 Label the vial with lab sample number, extraction date, matrix and analysis. Store extract vials at a temperature of 4 ± 2 °C until ready for analysis. Indicate in the extractions logbook the box number and "tray location" of the individual extract vials.

8 QUALITY CONTROL AND ACCEPTANCE CRITERIA

A method blank must be extracted for each and every item listed below:

- Each sample matrix (soil, water)
- Each day of extraction (24 hours midnight - midnight)
- Each extraction method or level
- Every 20 samples extracted in a 24-hour period

A laboratory control sample (LCS) is required for each and every item listed below:

- Each sample matrix
- Each extraction method or level
- Every extraction batch of twenty or fewer samples

Refer to the current revision of the applicable Katahdin SOP for analysis of extractable semivolatile organics for quality control acceptance criteria.

Each extractions analyst must demonstrate proficiency in performing the extractions that prepare samples for analysis. Demonstration consists of preparation (extraction), by the analyst, of at least four aliquots which are then analyzed according to the analytical method in question. These QC samples must meet all quality control acceptance limits. Demonstration must be documented by use of a form which summarizes the results of the analysis of these aliquots, calculated percent recoveries, and standard deviation. Demonstration of proficiency must be done one time per analyst initially and then annually thereafter. Refer to SOPs QA-805 and QA-807, current revision.

If, upon analysis of the extracted samples, it is discovered that quality control acceptance criteria have not been met, all associated samples must be evaluated against all the QC. In some cases data may be reported, perhaps with narration, while in other cases, other corrective action may be taken. The corrective actions may include re-extraction of the samples associated with the quality control sample that did not meet acceptance criteria, or may include making new reagents and standards if the standardization is suspect. These decisions are based on holding time considerations, client and project specific Data Quality Objectives and on review of chromatograms. The supervisor, Operations Manager, and/or Quality Assurance Officer may be consulted to evaluate data. Some samples may not be able to be reanalyzed within hold time. In these cases "qualified" data with narration may be advisable after consultation with the client.

Much of the work performed at the lab is analyzed in accordance with specific QC requirements spelled out in a project specific Quality Assurance Project Plan (QAPP) or in a program specific Quality Systems Manual (QSM). The reporting limits, acceptance criteria and/or corrective actions may be different than those specified in this SOP. In these cases the appropriate information will be communicated to the Department Manager and/or senior chemists before initiation of the analyses so that specific product codes can be produced for the project. In addition, the work order notes for each project will describe the specific QAPP or QSM to be followed.

9.0 METHOD PERFORMANCE

The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero. The MDLs are determined annually per type of instrument and filed with the Organic Department Manager and with the QAO. Refer to the current revision of Katahdin SOP QA-806, Method

Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications, for procedures on determining the MDL.

Refer to the applicable analytical SOP for other method performance parameters and requirements.

10.0 APPLICABLE DOCUMENTS/REFERENCES

Test Methods for Evaluating Solid Waste-Physical/Chemical Methods, Method 3540C, SW-846, Third Edition, Updates I, II, IIA, IIB, and III Revised December 1996, US EPA.

Department of Defense Quality Systems Manual for Environmental Laboratories (DOD QSM), Version 4.1, 04/22/09.

The National Environmental Laboratory Accreditation Conference (NELAC) Standards, June 2003.

LIST OF TABLES AND FIGURES

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SOXHLET EXTRACTION USING METHOD 3540 FOR SUBSEQUENT EXTRACTABLE SEMIVOLATILE ANALYSIS**

TABLE 1

SUMMARY OF METHOD MODIFICATIONS

TOPIC	KATAHDIN SOP CA-526-06	METHOD 3540, current revision
Apparatus/Materials	1. short stem funnels	2. drying columns
Reagents		
Sample preservation/ handling		
Procedures	1. Use 30 grams of sample and 30 grams of sodium sulfate 2. Place a plug of glass wool in soxhlet then add sample 3. Use 250 mL of methylene chloride for extraction 4. Extract the sample for 18 - 24 hours 5. Extract dried using Na_2SO_4 in short stem funnels 6. Rinse the extract flask three times with ~ 2 – 3 mLs of methylene chloride then rinse the sodium sulfate with ~ 15 mLs of methylene chloride to complete a quantitative transfer 7. no apparatus height specification for concentration on water bath 8. Water bath at 75-85 deg C 9. Sample removed from water bath when volume reaches ~6 mL	1. Use 10 grams of sample and 10 grams of sodium sulfate. 2. Place sample between 2 plugs of glass wool 3. Use 300 mL of methylene chloride for extraction 4. Extract the sample for 16 - 24 hours at 4 - 6 cycles/hour 5. Extract dried using Na_2SO_4 in drying columns 6. Wash the extractor flask and sodium sulfate column with 100 to 125 mL of extraction solvent to complete the quantitative transfer 7. partially immerse concentrator tube in water and lower apparatus to complete concentration in 10-20min 8. Water bath at 15-20 deg C above solvent boiling point 9. Sample removed from water bath when volume reaches 1-2 mL
QC - Spikes	1. Acid surrogate and spike components at 100 ug/mL; base/neutral surrogate and spike components at 50 ug/mL	1. Acid surrogate and spike components at 200 ug/mL; base/neutral surrogate and spike components at 100 ug/mL
QC - LCS	1. Acid surrogate and spike components at 100 ug/mL; base/neutral surrogate and spike components at 50 ug/mL	1. Acid surrogate and spike components at 200 ug/mL; base/neutral surrogate and spike components at 100 ug/mL
QC - Accuracy/Precision		
QC – MDL		

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SOXHLET EXTRACTION USING METHOD 3540 FOR SUBSEQUENT EXTRACTABLE SEMIVOLATILE ANALYSIS**

FIGURE 1

EXAMPLE OF LOGBOOK PAGE

SV SOX

KATAHDIN ANALYTICAL SERVICES, INC.

ORGANIC EXTRACTIONS LOG - SOIL SEMIVOLATILE

Extraction Method: (check one)	SW846 3550 (SONIC.)	SW846 3540 (SOX) ✓	SW846 3545 (ASE)
	SW846 3580 (OIL / WIPES)	OTHER	
Analytical Method: (check one)	SW846 8270 ✓	OTHER	
Standards	Surrogate ID (1): SV2324	Spike ID (1): SV2320	Spike ID (3):
	Surrogate ID (2):	Spike ID (2):	
Solvents / Chemicals / Consumables	Solvent Lot # (Mecl2): H18E08	Solvent Lot # (Acetone): —	Sodium Sulfate (granular) Lot # ~~2769~~ 2769002
	Filter Paper Lot # (SON) —	Filter Paper Lot # (KD) K11672363	Sodium Sulfate (powder) Lot # ~~2797~~ 2797001
Misc.	Nitrogen Bath Temperature: 35°C	Sonicator Horns Tuned: —	Balance ID: Mettler PJ400

QAEX169

Ext. Date	Ext. Init.	Sample ID	Initial Weight (g)	Surr. Vol. (mL)	Spk. Vol. (mL)	Fraction [illegible]	Fraction [illegible]	Pre-GPC Date Conc.	Pre-GPC Conc. Init.	Pre-GPC Final Vol. (mL)	Post-GPC Date Conc.	Post-GPC Conc. Init.	Post-GPC Final Vol. (mL)	Tray Loc.	Comments
6-23-09	KF	UG65386-1	30.00	1mL	NR	✓		6-26-09	KF	5mL	6-29-09	KF	1mL	SV451 B6	R102843
		-2	29.98		1mL									B7	
		-3	29.96											B8	
		GPC Blank	6-29-09											B9	
									KF	6-26-09					

0000007

Ext. Date	Ext. Init.	Sample ID	Initial Weight (g)	Surr. Vol. (mL)	Spk. Vol. (mL)	Fraction [illegible]	Fraction [illegible]	Pre-GPC Date Conc.	Pre-GPC Conc. Init.	Pre-GPC Final Vol. (mL)	Post-GPC Date Conc.	Post-GPC Conc. Init.	Post-GPC Final Vol. (mL)	Tray Loc.	Comments
6-23-09	KF	SC3258-11F	31.04	1mL	NR	✓		6-26-09	KF	5mL	6-30-09	GN	1mL	SV451 B10	
		-12F	30.02											C1	
		-13F	30.00											C2	
		-14F	30.01											C3	
		-15F	29.96											C4	
		-16F	30.05											C5	
		-17F	30.02											C6	
		-18F	29.99											C7	
		-19F	29.99											C8	
		-20F	30.00											C9	
		SC3259-3F	29.99											C10	
		-4F	29.96											D1	
		-5F	30.00											D2	
		SC3305-3F	30.01											D3	
									KF	6-26-09					

QAEX169

0000008

Reviewed By__________ Date__________

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SOXHLET EXTRACTION USING METHOD 3540 FOR SUBSEQUENT EXTRACTABLE SEMIVOLATILE ANALYSIS**

FIGURE 2

LCS/MATRIX SPIKE COMPONENT LIST

BASE/NEUTRALS	
1-Methylnaphthalene	Bis (2-chloroethoxy) methane
1,1-Biphenyl	Bis (2-chloroethyl) ether
1,2,4-Trichlorobenzene	Bis (2-Chloroisopropyl) ether)
1,2-Dichlorobenzene	Bis (2-ethylhexyl) adipate
1,3-Dichlorobenzene	Bis (2-ethylhexyl) phthalate
1,4-Dichlorobenzene	Butylbenzyl phthalate
1,4-Dioxane	Caprolactam
2,4-Dinitrotoluene	Carbazole
2,6-Dinitrotoluene	Chrysene
2-Chloronaphthalene	Dibenz (a, h) anthracene
2-Methylnaphthalene	Dibenzofuran
2-Nitroaniline	Diethyl adipate
3,3'-Dichlorobenzidine	Diethyl phthalate
3-Nitroaniline	Dimethyl phthalate
4-Bromophenylphenyl ether	Di-n-butylphthalate
4-Chloroaniline	Di-n-octyl phthalate
4-Chlorophenylphenyl ether	Fluoranthene
4-Nitroaniline	Fluorene
Acenaphthene	Hexachlorobenzene
Acenaphthylene	Hexachlorobutadiene
Acetophenone	Hexachlorocyclopentadiene
Aniline	Hexachloroethane
Anthracene	Indeno (1,2,3-cd) pyrene
Atrazine	Isophorone
Azobenzene	Naphthalene
Benzaldehyde	Nitrobenzene
Benzidine	N-Nitrosodimethylamine
Benzo (a) Anthracene	N-Nitroso-di-n-propylamine
Benzo (a) pyrene	N-Nitrosodiphenylamine
Benzo (b) fluoranthene	Phenanthrene
Benzo (ghi) perylene	p-toluidine
Benzo (k) fluoranthene	Pyrene
Benzyl alcohol	Pyridine

ACIDS		
2, 3, 4, 6-Tetrachlorophenol	2-Chlorophenol	Benzoic acid
2,4,5-Trichlorophenol	2-Methylphenol	Ethyl methanesulfonate
2,4,6-Trichlorophenol	2-Nitrophenol	Methyl methanesulfonate
2,4-Dichlorophenol	4,6-Dinitro-2-methylphenol	Pentachlorophenol
2,4-Dimethylphenol	4-Chloro-3-methylphenol	Phenol
2,4-Dinitrophenol	4-Methylphenol	
2,6-Dichlorophenol	4-Nitrophenol	

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SOXHLET EXTRACTION USING METHOD 3540 FOR SUBSEQUENT EXTRACTABLE SEMIVOLATILE ANALYSIS**

FIGURE 3

APPENDIX IX LCS/MATRIX SPIKE COMPONENT LIST

1,2,4,5-Tetrachlorobenzene	Hexachloropropene
1,3,5-Trinitrobenzene	Isodrin
1,4-Naphthoquinone	Isosafrole
1-Chloronaphthalene	Kepone
1-Naphthylamine	m-Dinitrobenzene
2,4-D	Methapyrilene
2-Acetyl aminofluorene	Methyl parathion
2-Naphthylamine	n-Nitrosodiethylamine
2-Picoline	n-Nitrosodi-n-butylamine
3,3-Dimethylbenzidine	n-Nitrosomethylethylamine
3-Methylcholanthrene	n-Nitrosomorpholine
4-Aminobiphenyl	n-Nitrosopyrrolidine
4-Nitroquinoline-1-oxide	n-Nitrotrosopiperidine
5-Nitro-o-toluidine	O,O,O-Triethyl phosphorothioate
7,12-Dimethylbenz(a)anthracene	o-Toluidine
a,a-Dimethylphenethylamine	Parathion
Acetophenone	p-Dimethylaminoazobenzene
Aramite	Pentachlorobenzene
Chlorobenzilate	Pentachloronitriobenzene
Diallate	Phenacetin
Dibenz(a,j)acridine	Phorate
Dimethoate	p-Phenylenediamine
Dinoseb	Pronamide
Diphenylamine	Safrole
Disulfoton	Silvex (2,4,5-TP)
Famphur	Sulfotep
Hexachlorophene	Thionazin

TITLE: PREPARATION OF SEDIMENT/SOIL AND TISSUE SAMPLES BY ACCELERATED SOLVENT EXTRACTION USING METHOD 3545 FOR SUBSEQUENT EXTRACTABLE PESTICIDE and PCB ANALYSIS

Prepared By: Galen Nickerson Date: 6-23-06

Approved By:

Department Manager: Peter Lee Date: 6-23-06

Operations Manager: Deborah J. Nadeau Date: 6.23.06

QA Officer: Leslie Dimond Date: 6-23-06

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
01	Sect. 1.4 - Added solvent and acid waste streams. Changed solvent in sect. 7.4.2 from hexane to CH_2Cl_2. Updated logbook example. Added recording of solvent, Na_2SO_4, hydromix, filterpaper Lot #'s as well as the temperature of the N_2 waterbath in the logbook.	LAD	07/08	07/08
02	Updated section 4 with current materials. Updated section 7 with current techniques. Changed when Hexane is added during the solvent exchange process. Changed all weights to record to 0.05g. Updated logbook example. Added CA-108 reference for subsampling.	LAD	08/09	08/09

TITLE: **PREPARATION OF SEDIMENT/SOIL AND TISSUE SAMPLES BY ACCELERATED SOLVENT EXTRACTION USING METHOD 3545 FOR SUBSEQUENT EXTRACTABLE PESTICIDE and PCB ANALYSIS**

Please acknowledge receipt of this standard operating procedure by signing and dating both of the spaces provided. Return the bottom half of this sheet to the QA Department.

I acknowledge receipt of copy _____ of document **SOP CA-537-02,** titled **Preparation of Sediment/Soil and Tissue Samples by Accelerated Solvent Extraction Using Method 3545 for Subsequent Extractable Pesticide and PCB Analysis**.

Recipient: _____ Date: _____

KATAHDIN ANALYTICAL SERVICES, INC.
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy _____ of document **SOP CA-537-02,** titled **Preparation of Sediment/Soil and Tissue Samples by Accelerated Solvent Extraction Using Method 3545 for Subsequent Extractable Pesticide and PCB Analysis**.

Recipient: _____ Date: _____

TITLE: **PREPARATION OF SEDIMENT/SOIL AND TISSUE SAMPLES BY ACCELERATED SOLVENT EXTRACTION USING METHOD 3545 FOR SUBSEQUENT EXTRACTABLE PESTICIDE and PCB ANALYSIS**

1.0 SCOPE AND APPLICATION

Method 3545 is a procedure for extracting water insoluble or slightly water soluble semivolatile organic compounds from soils, clays, sediments, sludges, waste solids and tissues samples. This Pressurized Fluid Extraction (PFE) method uses elevated temperature (100°C or 175°C) and pressure (1500 - 2000 psi) to achieve analyte recoveries equivalent to those from Soxhlet extraction, using less solvent and taking significantly less time than the Soxhlet procedure.

This method is applicable to the extraction of organochlorine pesticides and PCBs. Organochlorine pesticides and PCBs may then be analyzed by a variety of chromatographic procedures.

1.1 Definitions

ANALYTICAL BATCH: 20 or fewer samples which are analyzed together with the same method sequence and the same lots of reagents and with the manipulations common to each sample within the same time period or in continuous sequential time periods.

METHOD BLANK (laboratory reagent blank): An artificial sample designed to determine if method analytes or other interferences are present in the laboratory environment, the reagents, or the apparatus. For aqueous samples, reagent water is used as a blank matrix; for soil samples, baked organic-free sand is used as a blank matrix. The blank is taken through the appropriate steps of the process.

LABORATORY CONTROL SAMPLE (LCS): A blank that has been spiked with the analyte(s) from an independent source, and is analyzed exactly like a sample. Its purpose is to determine whether the methodology is in control, and whether the laboratory is capable of making accurate and precise measurements. The matrix used should be phase matched with the samples and well characterized.

MATRIX SPIKE/MATRIX SPIKE DUPLICATE (MS/MSD): Predetermined quantities of stock solutions of certain analytes are added to a sample matrix prior to sample extraction and analysis. Samples are split into duplicates, spiked and analyzed. Percent recoveries are calculated for each of the analytes detected. The relative percent difference between the samples is calculated and used to assess analytical precision.

SURROGATES: Organic compounds which are similar to analytes of interest in chemical composition, extraction and chromatography, but which are not normally found in environmental samples. These compounds are spiked into all blanks, standards, samples and spiked samples prior to analysis. Percent recoveries are calculated for each surrogate.

TITLE: **PREPARATION OF SEDIMENT/SOIL AND TISSUE SAMPLES BY ACCELERATED SOLVENT EXTRACTION USING METHOD 3545 FOR SUBSEQUENT EXTRACTABLE PESTICIDE and PCB ANALYSIS**

1.2 Responsibilities

This method is restricted to use by, or under the supervision of analysts experienced in the extraction of samples for pesticide and PCB analysis. Each analyst must demonstrate and document their ability to generate acceptable results with this method. Refer to Katahdin SOP QA-805, current revision, "Personnel Training".

It is the responsibility of all Katahdin technical personnel involved in the extraction of samples for pesticide and PCB analysis to read and understand this SOP, adhere to the procedures outlined, and to properly document their data in the appropriate lab notebook. Any deviations from the test or irregularities with the samples should also be recorded in the lab notebook and reported to the Department Manager or designated qualified data reviewer responsible for this data.

It is the responsibility of the Department Manager to oversee that the members of his/her group follow this SOP, to assure that their work is properly documented, and to indicate periodic review of the pertinent logbooks.

1.3 Safety

Users of this procedure must be cognizant of inherent laboratory hazards, proper disposal procedures for contaminated materials and appropriate segregation of hazardous wastes. The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical should be treated as a potential health hazard. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Everyone involved with the procedure must be familiar with the MSDSs for all the materials used in this procedure.

Each qualified analyst or technician must be familiar with Katahdin Analytical Environmental Health and Safety Manual including the Katahdin Hazardous Waste Management Plan and must follow appropriate procedures. These include the use of appropriate personal protective equipment (PPE) such as safety glasses, gloves, and lab coats when working with chemicals or near an instrument and not taking food or drink into the laboratory. Each analyst should know the location of all safety equipment. Each analyst shall receive a safety orientation from their department manager, or designee, appropriate for the job functions they will perform.

1.4 Waste Disposal

Wastes generated during the preparation of samples must be disposed of in accordance with the procedures described in the current revision of the Katahdin Hazardous Waste Plan and Safety Manual and SOP SD-903, "Sample Disposal," current revision. Expired standards are lab packed, placed in the Katahdin hazardous waste storage area, and disposed of in accordance with this SOP.

Any methylene chloride solvent waste generated during the rinsing of glassware etc. should be disposed of in the "D" waste stream satellite accumulation area nearest the point of generation. Acetone and hexane are considered flammable waste, and should be disposed of in the "O" waste stream satellite accumulation area nearest the point of generation. Post-extraction soil samples, used glass wool, and sodium sulfate waste should be disposed of in the soil with organics "I" waste stream satellite accumulation area nearest the point of generation. Acid waste generated during the cleanup of PCB samples should be disposed of in the "K" satellite accumulation area nearest the point of generation. Please refer to the current revision of SOP CA-107 for the location of satellite waste accumulation areas.

2.0 SUMMARY OF METHOD

Samples are prepared for extraction by weighing out a specific quantity of sample and mixing said quantity with pelletized diatomaceous earth (Hydromatrix) to remove moisture. The sample is then loaded into an extraction cell and placed on the Accelerated Solvent Extractor (ASE).

The extraction cell containing the sample is heated to the analyte-specific extraction temperature (see section 7.0), pressurized with hexane and extracted for a period of time outlined in section 7.0.

The solvent is collected from the extraction cells into 60 mL vials and allowed to cool. The solvent is then concentrated and, as needed, exchanged into solvent compatible with the cleanup or determinative step being employed.

3.0 INTERFERENCES

Contaminants in solvents, reagents, glassware, and other sample processing hardware may cause method interferences such as discrete artifacts and/or elevated baselines in the total ion current profiles (TICPs). All of these materials routinely must be demonstrated to be free from interferences under the conditions of the analysis by running laboratory method blanks. Interferences caused by phthalate esters can pose a major problem in semivolatile organics analysis because many phthalates are also target analytes. Common flexible plastics contain varying amounts of phthalates that are easily extracted during laboratory operations, so cross-

contamination of glassware frequently occurs when plastics are handled. Interferences from phthalates can best be minimized by avoiding the use of such plastics in the laboratory. At no time may gloves that have not been tested for phthalates or gloves known to contain phthalates be used or stored in the organic extraction lab. Additionally, whenever possible plastic items in this lab must be replaced with metal or Teflon or other non-phthalate plastic substitute.

Special care should be taken to ensure clean glassware and apparatus are used, pre-rinsed with the appropriate solvent prior to use. Solvents should be analyzed prior to use to demonstrate that each lot is free of contaminants that may interfere with the analysis. Interferences coextracted from the samples will vary considerably from source to source. If analysis of an extracted sample is prevented due to interferences, further cleanup of the sample extract may be needed to minimize interferences.

4.0 APPARATUS AND MATERIALS

4.1. Pressurized fluid extraction device: Dionex Accelerated Solvent Extractor, Model number ASE 200 with appropriately sized extraction cells. The cells will accommodate 15 g of sample and are made of stainless steel which is capable of withstanding the pressure requirements necessary for this procedure.

4.2. Analytical Balance: Mettler PJ4000 balance capable of weighing to 0.01 g

4.3. Vials for the collection of extracts: Dionex 60 ml pre-cleaned, open to screw cap with PTFE-lined silicone septum.

4.4. Filter disk for extraction vessels: Dionex 1.91 cm, Type D28.

4.5. 400 ml beakers

4.6 Ottawa Sand (Fisher P/N 523-3)

4.7 Kuderna-Danish (K-D) apparatus

4.7.1 Concentrator tube - 10-mL

4.7.2 Evaporation flask - 500-mL

4.7.3 Snyder column - Three-ball macro

4.8 Nitrogen evaporation (N-EVAP) apparatus.

4.9 Stainless steel spatula.

4.10 Porcelain mortar and pestle.

4.11 Boiling stones, 12 mesh silicon carbide (carborundum) – pre-purified by Soxhlet extraction in methylene chloride.

4.12 Vials - Glass, 12 mL and 4 mL capacity

4.13 500 ul syringes

4.14 Funnel powder, glass.

4.15 Filter paper, 18.5 cm, Fisherbrand or Whatman 114V (or equivalent)

4.16 Water bath or steam bath capable of maintaining a temperature of at least 85°C.

5.0 REAGENTS

5.1 Aluminum oxide (Alumina, acid), Brockman activity I, 60-325 mesh

5.2 Drying agents

5.2.1 Pelletized diatomaceous earth (Hydromatrix) - Fisher P/N.

5.2.2 Sodium sulfate crystals, Na_2SO_4.

5.3 Extraction solvents

5.3.2 Methylene Chloride - Pesticide grade or equivalent. Lot must be verified by concentrating ≈ 300 mL to 1 mL and evaluating by both GC/MS and GC/FID analyses.

5.3.3 Hexane - Pesticide grade or equivalent. Lot must be verified by concentrating 200-300 mLs to 1 mL followed by GC/ECD analysis.

5.3.4 Acetone – Pesticide grade or equivalent. Lot must be verified by concentrating ≈ 300 mL to 1 mL followed by GC/MS, GC/ECD, and GC/FID analyses.

5.3.5 Organochlorine pesticides and PCBs may be extracted with acetone/methylene chloride (1:1,v/v) or hexane (100%).

5.3.6 Other solvent systems may be employed, provided that the analyst can demonstrate adequate performance for the analytes of interest in the sample matrix

CAUTION: For best results with very wet samples (e.g., ≥ 30% moisture), reduce or eliminate the quantity of hydrophilic solvent used.

5.4 Pre-purified nitrogen is used to purge and/or pressurize the extraction cell.

5.5 Spiking Solutions

5.5.1 Pesticide/PCB surrogate spiking solution - Prepare a solution of decachlorbiphenyl (DCB) and tetrachloro-meta-xylene (TCMX) at a concentration of 1 ug/mL in acetone. Store the solution at –10 to -20 °in a Teflon sealed container. Solution must be verified by GC/ECD prior to use, and must be replaced every 6 months or sooner if degradation is evident.

5.5.2 Pesticide/PCB matrix spike/Lab control sample spiking solution - Prepare separate spiking solutions for Pesticides and one for PCBs in pesticide grade methanol and acetone, respectively, that contain all target analytes listed below:

Pesticide Spiking Solution

Analyte	ug/mL	Analyte	ug/mL
4,4'-DDD	0.5	Endosulfan I	0.5
4,4'-DDE	0.5	Endosulfan II	0.5
4,4'-DDT	0.5	Endosulfan Sulfate	0.5
Aldrin	0.5	Endrin	0.5
alpha-BHC	0.5	Endrin Aldehyde	0.5
beta-BHC	0.5	Endrin Ketone	0.5
delta-BHC	0.5	gamma-BHC (Lindane)	0.5
Dieldrin	0.5	Heptachlor	0.5
alpha-Chlordane	0.5	Heptachlor Epoxide	0.5
gamma-Chlordane	0.5	Methoxychlor	0.5

PCB Spiking Solution

Analyte	ug/mL
Aroclors 1016/1260	5.0

Store the solution at –10 to -20 °C in a Teflon sealed container. The solution must be verified by GC/ECD prior to use, and must be replaced every 6 months or sooner if degradation is evident.

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

TITLE: **PREPARATION OF SEDIMENT/SOIL AND TISSUE SAMPLES BY ACCELERATED SOLVENT EXTRACTION USING METHOD 3545 FOR SUBSEQUENT EXTRACTABLE PESTICIDE and PCB ANALYSIS**

Holding time for extraction of sediment/soil and tissue samples for Method 3545 is 14 days from date of sample collection, although the analyst should be aware that actual holding times employed may be project/program specific.

Store all extracts at 4°C (± 2°C) in the dark in labeled Teflon-sealed containers. See SOP SD-902, "Sample Receipt and Internal Control," current revision, for storage areas and temperature maintenance procedures.

7.0 PROCEDURES

7.1 Preparing the Accelerated Solvent Extractor

7.1.1 Before extractions can begin, the system must first go through its daily maintenance checks. To begin, turn on the system and nitrogen gas tank. Check the system pressure to ensure that the solvent pressure is at 10 psi, air is at 50 psi, and compression is at 130 psi. Next, check the solvent bottle and fill with the appropriate solvent if necessary. At this time the rinse and waste vials should be emptied and four 60 ml vials need to be placed in the rinse positions of the sample collection carousel. Record the solvent lot number in the extraction logbook.

7.1.2 The system must now be rinsed before the analysis can begin. Press the rinse button on the touch pad. The system will immediately begin its rinse cycle. This rinse procedure should be repeated at least three times before any samples are extracted to ensure that all contaminants are removed from the system.

7.1.3 To prepare extraction cells, disassemble the ASE extractor cells and clean each piece with soapy water. Next rinse each piece with acetone to ensure that all water is removed. Next, rinse the body and two screw caps three times with methylene chloride. Be sure you are using a solvent whose lot has been checked. Inspect each screw cap to ensure that the o-rings are not crushed and in need of replacement. O-rings should be replaced after approximately 50 extractions to ensure proper fit. Cell bodies should also be checked for nicks and dings at each end. If any dings are present the cell should be placed out of rotation until it can be repaired. If any instrument maintenance is necessary record the maintenance performed in the instrument maintenance logbook.

7.1.4 Once the extraction cells have been rinsed replace the bottom cell screw cap. The bottom of the cell body is the end that is imprinted with Dionex.

Next, place 2 19.8 mm filter disks into the top of the cell body and push it to the bottom of the cell with the plunger that was supplied with the system. Record the lot numbers of the filter disks in the extraction logbook.

If extracting pesticides/PCBs from tissue samples weigh out 5.00 ±1.00 g of aluminum oxide (alumina, acid) into cell and place a second filter on the top of alumina. Avoiding matrix affect interference for tissue sample, the aluminum oxide is used to remove lipid. Record the lot numbers of the aluminum oxide and filters in the extraction logbook.

7.1.5 Label each extraction cell and clean, rinsed, 60 ml collection vial with the appropriate sample ID with a black marker.

7.2 Sample preparation

7.2.1 Sediment/soil samples - Decant and discard any water layer on a sediment sample. Mix the sample thoroughly with the stainless steel spatula. If the sample container is full to the extent that stirring the sample is impractical, try to remove the “best representative” aliquot from the jar based on color, particle size, moisture, etc. Discard any foreign objects such as sticks, leaves, and rocks.

7.2.2 Gummy, fibrous, or oily materials not amenable to grinding should be cut, shredded, or otherwise reduced in size to allow mixing and maximum exposure of the sample surfaces for the extraction. Materials such as glass, rubber, metal, etc. may not require mixing with Hydromatrix to disperse the sample. Plastic materials must be tested for degradation (melting) in methylene chloride prior to ASE extraction.

7.2.3 Tissue samples are blended prior to handling in extraction laboratory.

7.2.4 Refer to Katahdin Analytical Services SOP CA-108, current revision, “Basic Laboratory Technique” for more information regarding subsampling.

7.3 Sample Handling

The following steps should be performed rapidly to avoid loss of the more volatile extractables.

7.3.1 Weigh out 15.00 ± 0.05g portion of sample into a 400 ml beaker. For tissue sample, weigh out a 5.00 ± 0.05g portion of sample into a 400 ml beaker.

Record sample weight to the nearest 0.05g in the appropriate extraction logbook.

7.3.2 Add enough Hydromatrix to the sample to produce a "free-flowing" mixture, around 5g. For tissue sample, pulverize the mixture (sample-hydromatrix) using the porcelain mortar-pestle apparatus. At this point don't add an excessive amount because more Hydromatrix can be added later in the procedure. This ensures that the sample will properly fit in the extraction cell. The amount of Hydromatrix will depend upon the moisture content of the sample (e.g., low moisture content will require less Hydromatrix). Mix well with a spatula. Keep the spatula in the sample beaker and cover the beaker with aluminum foil. Record the Hydromatrix lot number in the extraction logbook.

7.3.3 A method blank must be prepared with each extraction batch, not to exceed 20 client samples. To prepare a method blank, weigh out a 5.00 ± 0. 05g portion of Ottawa sand for tissue sample batch or 15.00 ± 0.05g portion for sediment/soil sample batch into a 400 ml beaker. Record the weight to the nearest 0.05 g in the appropriate extraction logbook. Add enough Hydromatrix to the sand to produce a "free-flowing" mixture. At this point don't add an excessive amount because more Hydromatrix can be added later in the procedure. This ensures that the sample will properly fit in the extraction cell. Mix well with a spatula. Keep the spatula in the sample beaker and cover the beaker with aluminum foil.

7.3.4 A laboratory control sample (LCS) must be prepared with each extraction batch, not to exceed 20 client samples. If no MS/MSD is to be prepared a laboratory control sample duplicate must be prepared. To prepare an LCS (and LCSD), weigh out a 15.00 ± 0.05g portion of Ottawa sand into a 400 ml beaker; for tissue sample, weigh out a 5.00 ± 0. 05 g of Ottawa sand into a 400 mL beaker. Record the weight to the nearest 0. 05 g in the appropriate extraction logbook. Add enough Hydromatrix, approximately 5g to the sand to produce a "free-flowing" mixture. At this point don't add an excessive amount because more Hydromatrix can be added later in the procedure. This ensures that the sample will properly fit in the extraction cell. Mix well with a spatula. Keep the spatula in the sample beaker and cover the beaker with aluminum foil. **If extracting both pesticide and PCB separate LCS/D and MS/D need to be prepared.**

7.3.5 A matrix spike/matrix spike duplicate (MS/MSD) set must be prepared for every 20 samples. To prepare MS/MSD, weigh out a 15.00 ± 0.05g portion of designee sample into a 400 ml beaker. For tissue sample, weigh out a 5.0 ± 0.05g portion of designee sample into a 400 mL beaker. Record the

weight to the nearest 0.05g in the appropriate extraction logbook. Add enough Hydromatrix approximately 5g to the sand to produce a "free-flowing" mixture. At this point don't add an excessive amount because more Hydromatrix can be added later in the procedure. This ensures that the sample will properly fit in the extraction cell. Mix well with a spatula. Keep the spatula in the sample beaker and cover the beaker with aluminum foil.

7.3.6 Once all the QC and field samples have been weighed and mixed with a portion of Hydromatrix, find the appropriately labeled extraction cell and place the cell funnel, rinsed three times in methylene chloride, on the open end. This funnel was supplied with the system. Pour the contents of the beaker into the extraction cell. Carefully scrape all the mixtures from the beaker walls so that no more than 1% remains behind in the beaker. If there is head space remaining in the extraction cell fill the remaining space with more Hydromatrix by pouring the sample back into the beaker and mixing the new Hydromatrix into the sample. This will minimize the amount of solvent used. Again, pour the sample back into the same extraction cell and cover with a screw cap. Rinse the cell funnel with extraction solvent between each sample.

7.3.7 To all tissue samples, method blank, LCS, and MS/MSD add 0.2 mL of Pest/PCB Surrogate spiking solution using a pre-rinsed 500 uL gas tight syringe. To all sediment/soil samples and QC being extracted add 0.5 mL of Pest/PCB surrogate spiking solution using a pre-rinsed 0.5 mL gas-tight syringe. Record surrogate spike volume and identification code in extraction logbook. Thoroughly rinse syringe with solvent when spiking is complete. To LCS and MS add 0.5 mL of appropriate spike solution to each.

7.3.8 Replace all screw caps and ensure that each is properly tightened. Place each extraction cell into the ASE sample carousel. Place the corresponding collection vial into the appropriate location on the sample collection carousel. Be certain that the cell and vial are in the same number position of the carousel. In order to avoid a possible carryover, samples should be placed in order of their cleanness.

7.3.9 Press the menu button on the ASE touch pad. Choose option number one and press enter. Select method number one for Pesticide and PCB analysis, press enter. Press start on the
touch pad. The extraction process will begin and takes approximately 14 minutes per sample to complete.

NOTE: Recommended extraction conditions:

For Pest/PCB (saved in the instrument as method one)
Oven temperature: 100°C
Pressure: 1500 - 2000 psi
Static time: 5 min (after 5 min pre-heat equilibration)
Flush volume: 60% of the cell volume
Nitrogen purge: 90 sec at 150 psi (purge time may be extended for larger cells)
Static Cycles: 1

Optimize the conditions, as needed, according to the manufacturer's instructions. In general, the pressure is not a critical parameter, as the purpose of pressurizing the extraction cell is to prevent the solvent from boiling at the extraction temperature and to ensure that the solvent remains in intimate contact with the sample. Any pressure in the range of 1500 - 2000 psi should suffice.

Once established, the same pressure should be used for all samples extracted for the same analysis type.

As stated above, the recommended conditions have been saved as methods on the instrument. If it becomes necessary to re-program conditions, follow the above guidelines.

7.3.10 Once the extraction process is complete and the instrument is idle remove the sample extracts and label with appropriate sticker. They are now ready to be stored in the interim extract storage refrigerator unless the samples are to be concentrated the same day.

7.3.11 Remove the extraction cells from the ASE and empty their contents into the appropriate solid waste container.

7.3.12 The extract is now ready for concentration, cleanup, or analysis, depending on the extent of interferents and the determinative method to be employed. Certain cleanup and/or determinative methods may require a solvent exchange prior to cleanup and/or sample analysis.

7.4 Concentration of Extracts

7.4.1 If samples are to be GPC'd, refer to the current revision of Katahdin SOP CA-513, Extract Cleanup Using Gel Permeation Chromatography, for appropriate concentrating procedures.

7.4.2 If samples are not to be GPC'd follow steps 7.4.3 through 7.4.9 to concentrate extracts to final volume of 5.0 mLs for soil/sediment samples and 2.0 mL for tissue samples

7.4.3 Before assembling, rinse the K-D glassware (flask, concentration tube, and Snyder column, including the ground glass joints on the flask and columns) three times with hexane if using 100 % hexane for Pesticide/PCB extraction, if not rinse three times with methylene chloride. Add two boiling chips to the K-D. Insert 18.5 cm filter papers into short stem powder funnels and add ≈ 2 inches of sodium sulfate crystals. Rinse the sodium sulfate in the assembled funnels with ≈20-30 mLs of methylene chloride. Place the assembled K-D's under the funnels. Visually examine each 60mL vial of sample. If there is excessive moisture in the sample, biphasic layering will be seen. If this occurs, add approximately 1 inch of sodium sulfate crystals to the vial. This should remove most of the moisture. Record the lot number of the sodium sulfate in the extraction logbook.

7.4.4 For a solvent exchange, no GPC, add approximately 50 mL Hexane to funnel and let drain through. Since methylene chloride has a lower boiling point than Hexane, this will result in a final extract in hexane only. Record the lot number of the solvent in the extraction logbook.

7.4.5 Transfer the extracts to the K-D concentrator setups through the sodium sulfate in the funnels. This is the drying step, which is required to remove residual water from the extracts. After pouring all of the extract volume through the sodium sulfate, rinse the extract vial three times with methylene chloride. Add the rinsings through the sodium sulfate to complete a quantitative transfer. Rinse the sodium sulfate with ≈ 15 mLs of methylene chloride and allow draining.

7.4.6 Transfer the labels from the extract flasks to the K-Ds. Remove the funnel, add one or two clean boiling stones to the K-D evaporative flask and attach a 3-ball macro Snyder column. Pre-wet the Snyder column with 1 mL of hexane.

7.4.7 Place the K-D in a hot water bath (85-90°C). Gently swirl K-D in the water until boiling begins. At the proper distillation rate, the Snyder balls should chatter but the chambers should not flood with condensed solvent. The K-D should be kept in a vertical orientation while on the bath. When the apparent volume in the concentrator tube reaches ≈ 6 mL, remove the K-D from the water bath. Allow the K-D to cool for 10 minutes. Rinse the Snyder column lower joint with ≈ 1 mL of hexane. Remove the Snyder column. Wipe off

any water from the neck above the lower joint of the flask. Separate the K-D flask from the concentrator tube, rinsing the ground glass joint with ≈ 1 mL hexane.

7.4.8 Reduce the extract in the concentrator tube to approximately 1 mL using the nitrogen blow-down apparatus. The bath temperature must be no higher than the boiling point of the solvent (39°C for methylene chloride). Turn the gas to 3 psi. Be careful not to splash the extract out of the tube. During concentration on the N-evap, the internal wall of the concentrator tube and the N-evap sparging pipet must be rinsed down at least once or twice with ≈1 mL of hexane. The solvent level in the concentrator tube must be positioned below the level of the water bath as much as possible to prevent water from condensing into the sample extract. As the extract volume is reduced, lower the N_2 sparging pipet closer to the surface of the extract to expedite the concentration. Record the temperature of the water in the nitrogen evaporation water bath in the extraction logbook, also note any problems or extract losses, if they occur.

7.4.9 Complete quantitative transfer of the extract to a vial by using hexane. Adjust the volume of the hexane extract to 5 mL in a 12mL vial using the appropriate “reference vial” for volume comparison. For Pesticides/PCB from tissue samples, adjust the volume of hexane extract to 2.0 mL in a 4.0 mL vial using the appropriate “reference vial” for volume comparison.

7.4.10 Label the vial with lab sample number, extraction date, matrix and analysis. Store extract vials at a temperature of 4 ± 2 °C until ready for analysis. Indicate in the extractions logbook the box number and “tray location” of the individual extract vials.

7.4.11 All sample extracts for 8082 PCB analysis must undergo a sulfuric acid wash (cleanup) prior to analysis. Sample extracts for 8081 pesticide do not undergo further cleanup unless requested by the client. All soil/sediment sample extracts for combined 8081/8082 analyses must be split. One portion must be acid cleaned for 8082 analysis. The associated method blank must be split and acid-cleaned in the same fashion. PCB LCSs and matrix spikes are acid cleaned also. Pesticide LCSs and matrix spike samples are not subjected to further cleanup. Please refer to Katahdin SOP CA525 (current revision), Extract Cleanup Using Sulfuric Acid, for further instructions.

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

A method blank must be extracted for each and every item listed below:

- Each sample matrix (soil, water)
- Each day of extraction (24 hours midnight - midnight)
- Each extraction method or level
- Every 20 samples extracted in a 24-hour period

A laboratory control sample (LCS) is required for each and every item listed below:

- Each sample matrix
- Each extraction method or level
- Every extraction batch of twenty or fewer samples

Refer to the current revision of the applicable Katahdin SOP for analysis of Pesticides and PCBs for quality control acceptance criteria.

9.0 METHOD PERFORMANCE

The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero. The MDLs are determined annually per type of instrument and filed with the Organic Department Manager and with the QAO. Refer to the current revision of Katahdin SOP QA-806, Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications, for procedures on determining the MDL.

Refer to the applicable analytical SOP for other method performance parameters and requirements.

10.0 APPLICABLE DOCUMENTS/REFERENCES

Test Methods for Evaluating Solid Waste-Physical/Chemical Methods, Method 3545A, SW-846, Third Edition, Updates I, II, IIA, IIB, III, IIIA, IIIB and IV, Revised February 2007, US EPA.

Department of Defense Quality Systems Manual for Environmental Laboratories (DOD QSM), Version 4.1, 04/22/09.

The National Environmental Laboratory Accreditation Conference (NELAC) Standards, June 2003.

TITLE: **PREPARATION OF SEDIMENT/SOIL AND TISSUE SAMPLES BY ACCELERATED SOLVENT EXTRACTION USING METHOD 3545 FOR SUBSEQUENT EXTRACTABLE PESTICIDE and PCB ANALYSIS**

LIST OF TABLES AND FIGURES

TITLE: **PREPARATION OF SEDIMENT/SOIL AND TISSUE SAMPLES BY ACCELERATED SOLVENT EXTRACTION USING METHOD 3545 FOR SUBSEQUENT EXTRACTABLE PESTICIDE and PCB ANALYSIS**

TABLE 1

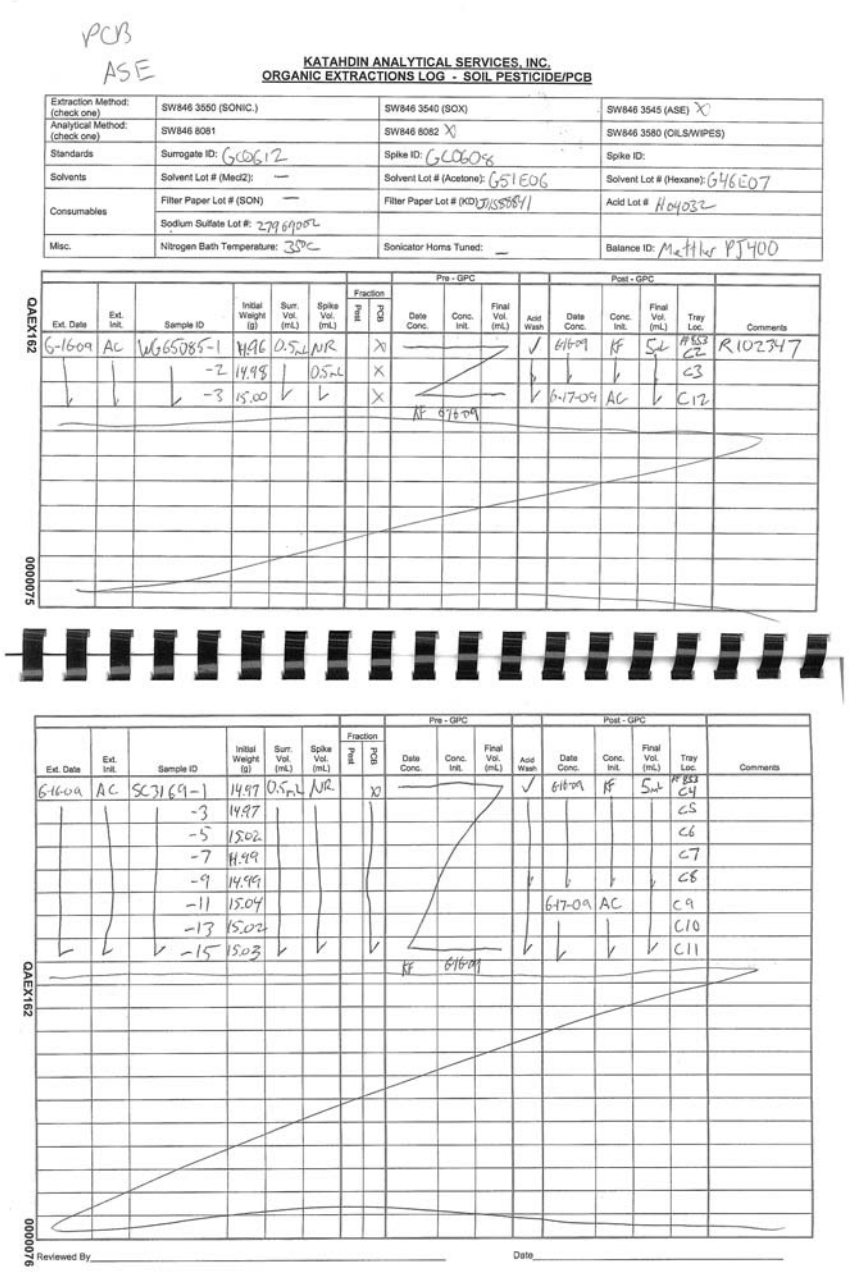
SUMMARY OF METHOD MODIFICATIONS

TOPIC	KATAHDIN SOP CA-537-02	METHOD 3545, current revision
Apparatus/Materials		
Reagents	Katahdin receives a certificate with each lot indicating sodium sulfate crystals were dried by the manufacturer at prescribed conditions.	Section 5.3.3 Drying agents should be purified by heating at 400 °C for 4 hours…
Sample preservation/ handling	Samples are not ground prior to mixing with the drying agent.	Section 7.3 Grind a sufficient weight of the dried sample…
Procedures	Section 7.3.9 Nitrogen purge for Pesticide/PCB and DRO: 90 sec at 150 psi Oven temperature for DRO: 175°C	Section 7.8 Recommended extraction conditions for all extractions: Nitrogen purge: 60 sec at 150 psi. Oven temperature: 100 °C
QC - Spikes		
QC - LCS		
QC - Accuracy/Precision		
QC - MDL		

TITLE: **PREPARATION OF SEDIMENT/SOIL AND TISSUE SAMPLES BY ACCELERATED SOLVENT EXTRACTION USING METHOD 3545 FOR SUBSEQUENT EXTRACTABLE PESTICIDE and PCB ANALYSIS**

FIGURE 1

EXAMPLE OF LOGBOOK PAGE



PCB
ASE

KATAHDIN ANALYTICAL SERVICES, INC.
ORGANIC EXTRACTIONS LOG - SOIL PESTICIDE/PCB

Extraction Method: (check one)	SW846 3550 (SONIC.)	SW846 3540 (SOX)	SW846 3545 (ASE) X
Analytical Method: (check one)	SW846 8081	SW846 8082 X	SW846 3580 (OILS/WIPES)
Standards	Surrogate ID: GC0612	Spike ID: GC0605	Spike ID:
Solvents	Solvent Lot # (Med2): —	Solvent Lot # (Acetone): G51E06	Solvent Lot # (Hexane): G46E07
Consumables	Filter Paper Lot # (SON) —	Filter Paper Lot # (KD) JI155581	Acid Lot # H04032
	Sodium Sulfate Lot #: 279690SL		
Misc.	Nitrogen Bath Temperature: 35°C	Sonicator Horns Tuned: —	Balance ID: Mettler PJ400

Ext. Date	Ext. Init.	Sample ID	Initial Weight (g)	Surr. Vol. (mL)	Spike Vol. (mL)	Fraction Prel	Fraction PCB	Pre-GPC Date Conc.	Pre-GPC Conc. Init.	Pre-GPC Final Vol. (mL)	Acid Wash	Post-GPC Date Conc.	Post-GPC Conc. Init.	Post-GPC Final Vol. (mL)	Tray Loc.	Comments
6-16-09	AC	WG65085-1	14.96	0.5mL	NR		X				✓	6/16/09	KF	5mL	#553 C2	R102347
		-2	14.98		0.5mL		X								C3	
		-3	15.00				X					6-17-09	AC		C12	
								KF 6/16/09								

QAEX162 0000075

Ext. Date	Ext. Init.	Sample ID	Initial Weight (g)	Surr. Vol. (mL)	Spike Vol. (mL)	Fraction Prel	Fraction PCB	Pre-GPC Date Conc.	Pre-GPC Conc. Init.	Pre-GPC Final Vol. (mL)	Acid Wash	Post-GPC Date Conc.	Post-GPC Conc. Init.	Post-GPC Final Vol. (mL)	Tray Loc.	Comments
6-16-09	AC	SC31691-1	14.97	0.5mL	NR		X				✓	6/16/09	KF	5mL	#553 C4	
		-3	14.97												C5	
		-5	15.02												C6	
		-7	14.99												C7	
		-9	14.99												C8	
		-11	15.04									6/17/09	AC		C9	
		-13	15.02												C10	
		-15	15.03					KF 6/16/09							C11	

QAEX162 0000076

Reviewed By_____ Date_____

TITLE: **ACID DIGESTION OF AQUEOUS SAMPLES BY EPA METHOD 3010 FOR ICP AND ICP-MS ANALYSIS OF TOTAL OR DISSOLVED METALS**

Prepared By: George Brewer Date: 11/97

Approved By:

Group Supervisor: George Brewer Date: 01/19/01

Operations Manager: John C. Buxton Date: 1/22/01

QA Officer: Deborah J. Nadeau Date: 1·22·01

General Manager: Dennis F. Keefe Date: 1/22/01

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
01 3010A	Format changes, added pollution prevention, block digester; revised database references; revised and added tables.	DN	1·22·01	1/22/01
02 3010A	Added wording allowing use of digestates for ICP-MS analysis. Added use of block digester as primary heating source & adjusted volumes. Revised standard solution names & concs. in Figures 3 & 4.	DN	8·29·02	8·29·02
03	Added Uranium to spiking solutions for LCS & MS/D. Removed the Internal Custody Record for Metals Digestates figure and reference.	LAD	04/06	04/06
04	Minor changes to Section 7 to reflect current practices. Updated Figure 1 - Sample Prep Logbook. Updated Figure 2 and 3 - Spike amounts.	LAD	05/09	05/09
05	Added references. Updated Figure 2 and 3 with correct spike information. Added CA-108 reference for subsampling information.	LAD	04/10	04/10

TITLE: **ACID DIGESTION OF AQUEOUS SAMPLES BY EPA METHOD 3010 FOR ICP AND ICP-MS ANALYSIS OF TOTAL OR DISSOLVED METALS**

Please acknowledge receipt of this standard operating procedure by signing and dating both of the spaces provided. Return the bottom half of this sheet to the QA Department.

I acknowledge receipt of copy ____ of document **SOP CA-604-05**, titled **ACID DIGESTION OF AQUEOUS SAMPLES BY EPA METHOD 3010 FOR ICP AND ICP-MS ANALYSIS OF TOTAL OR DISSOLVED METALS**.

Recipient: _____ Date: _____

KATAHDIN ANALYTICAL SERVICES, INC.
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy ____ of document **SOP CA-604-05**, titled **ACID DIGESTION OF AQUEOUS SAMPLES BY EPA METHOD 3010 FOR ICP AND ICP-MS ANALYSIS OF TOTAL OR DISSOLVED METALS**.

Recipient: _____ Date: _____

TITLE: **ACID DIGESTION OF AQUEOUS SAMPLES BY EPA METHOD 3010 FOR ICP AND ICP-MS ANALYSIS OF TOTAL OR DISSOLVED METALS**

1.0 SCOPE AND APPLICATION

The purpose of this SOP is to describe the procedure utilized by Katahdin Analytical Services, Inc. personnel to solubilize metals in aqueous samples, wastes that contain suspended solids, and mobility-procedure extracts prior to analysis by inductively coupled plasma atomic emission spectroscopy (ICP) and inductively coupled plasma mass spectrometry (ICP-MS). This SOP applies to samples prepared by EPA Method 3010, with the method modifications mentioned in Table 2.

1.1 Definitions - none.

1.2 Responsibilities

This method is restricted to use by, or under the supervision of analysts experienced in the acid digestion of aqueous samples by EPA Method 3010. Each analyst must demonstrate and document their ability to generate acceptable results with this method. Refer to Katahdin SOP QA-805, current revision, “Personnel Training & Documentation of Capability”.

It is the responsibility of all Katahdin technical personnel involved in the acid digestion of aqueous samples using EPA Method 3010 to read and understand this SOP, to adhere to the procedures outlined, and to properly document their work in the appropriate lab notebook. Any deviations from the method or irregularities with the samples should also be recorded in the lab notebook and reported to the Supervisor or designated qualified data reviewer responsible for these data.

It is the responsibility of the Supervisor to ensure that technical personnel perform acid digestions in accordance with this SOP and to confirm that their work is properly documented through periodic review of the associated logbooks.

1.3 Safety

The acids used in this procedure are highly corrosive and reactive, and spiking standards contain toxic metals. The toxicity and reactivity of client samples are usually unknown, so samples should always be assumed to present a contact hazard. To reduce or eliminate exposure to potentially harmful chemicals, lab coats, gloves, and safety glasses or goggles must be worn whenever handling samples or reagents. Additional safety apparel, including face shields, rubber aprons, dust masks, and rubber shoe protectors, is available in the metals prep lab and should be worn whenever circumstances warrant.

Acids should be added to samples slowly and carefully while watching for reactions. This should be done under a hood, in case harmful fumes are evolved.

Hood sashes should be lowered as far as possible whenever beakers are being heated in the hood. Use caution when handling hot beakers.

Each qualified analyst or technician must be familiar with Katahdin Analytical Health and Safety Manual including the Katahdin Hazardous Waste Management Plan and must follow appropriate procedures. These include the use of appropriate personal protective equipment (PPE) such as safety glasses, gloves and lab coats when working with chemicals or near an instrument and not taking food or drink into the laboratory. Each analyst should know the location of all safety equipment. Each analyst shall receive a safety orientation from their supervisor, or designee, appropriate for the job functions they will perform.

1.4 Pollution Prevention/Waste Disposal

Whenever possible, laboratory personnel should use pollution prevention techniques to address their waste generation. Refer to the current revision of the Katahdin Hazardous Waste Management Program for further details on pollution prevention techniques.

Excess spiking solutions must be emptied into the corrosive waste carboy located in the metals prep lab for subsequent appropriate disposal in accordance with the Chemical Hygiene Plan and Safety Manual.

Sample digestates should be stored for a minimum of 60 days after digestion to allow for analysis, and reanalysis if necessary. Digestates older than 60 days may be emptied into the corrosive waste carboy in the metals prep lab for subsequent appropriate disposal in accordance with the Chemical Hygiene Plan and Safety Manual.

Any other wastes generated during the preparation of samples must be disposed of in accordance with the Katahdin Chemical Hygiene Plan and Safety Manual and SOP SD-903, “Sample Disposal,” current revision.

2.0 SUMMARY OF METHOD

The aqueous sample is refluxed with nitric acid in a covered digestion vessel. Additional nitric acid is added until the color of the digestate has stabilized. After the digestate has been evaporated to a low volume, it is refluxed with hydrochloric acid and diluted to the appropriate final volume with reagent water.

Samples may be concentrated (i.e. final digestate volume less than initial sample volume) during digestion if lower detection limits are required. Volumes of reagents and spiking standards must be added in proportion to the final volume of the digestate. Because concentration of samples during digestion increases the concentrations of dissolved solids

and may exacerbate analytical interferences, concentration factors greater than 5 are not recommended.

3.0 INTERFERENCES

Interferences are discussed in the applicable analytical SOPs.

4.0 APPARATUS AND MATERIALS

4.1. 250 mL and 400 mL pre-cleaned Griffin beakers (cleaned according to the current revision of SOP CA-100, "Labware Cleaning") for digestion using a hot plate. If digestion will be performed using a block digester, 70ml graduated, polyethylene block digester tubes (with attached snap caps) will be used instead of glass beakers.

4.2 Ribbed watch glasses. If digestion is performed using a hot plate, 75 mm diameter and 100 mm diameter glass watch glasses (pre-cleaned as above) are used. If digestion is performed using a block digester, 40mm diameter disposable polyethylene watch glasses are used.

4.3 Adjustable volume automatic pipets covering the range from 10 uL to 1000 uL and disposable pipet tips; calibrated Finn pipets or Eppendorf pipets are acceptable.

4.4 Disposable graduated polystyrene specimen containers with pouring lips, 200 mL capacity.

4.5 Hot plate, block digester, or other heating source - adjustable and capable of maintaining a temperature of 90-95 ^{o}C. Hot plates must be numbered for easy identification.

4.6 Device for measuring hot plate temperature. This may consist of a heat-resistant 100ml beaker containing reagent water in which a thermometer is immersed. When using a block digester, a digestion tube containing reagent water in which a thermometer is immersed may be used. The temperature of one hot plate is measured each day, on a rotating basis. The hot plate identification number and the measured temperature are recorded on the sample preparation logbook sheet.

4.7 Plastic funnels, pre-cleaned as in Section 4.1.

4.8 Filter funnel holders, capable of suspending plastic funnels above disposable specimen containers.

4.9 Polyethylene wash bottles for dispensing reagent water and 5% HNO_3.

4.10 Filter paper, Whatman No. 41 or equivalent. Filters are acid-washed immediately prior to use as follows. Place a pre-cleaned funnel in the funnel holder and put a disposable plastic specimen container under the funnel to collect the rinsates. Place a folded filter in the funnel and rinse three times with approximate 10 mL volumes of 5% HNO_3, making sure the entire surface of the filter is wetted each time and allowing each rinse to drain completely before continuing. Then rinse three times with approximate 25 mL volumes of reagent water. Discard the rinsates into the appropriate waste container. The acid-washed filter is now ready for use.

4.11 Polyethylene sample containers with screw caps or graduated polyethylene sample containers with attached snap lids, 125 mL capacity. These are not necessary when using the block digester since the final digestates are stored in the digestion tubes.

4.12 Repipetters (adjustable repeating pipetters with reservoirs) for dispensing concentrated nitric acid and 1:1 HCl.

5.0 REAGENTS

5.1 Concentrated nitric acid, HNO_3 – trace metals grade.

5.2 Concentrated hydrochloric acid, HCl – trace metals grade.

5.3 Reagent water - water that meets the performance specifications of ASTM Type II water (ASTM D1193).

5.4 Hydrochloric acid, 1:1. Add a volume of concentrated hydrochloric acid to an equivalent volume of reagent water and swirl gently to mix.

5.5 Nitric acid, 5% v/v. Add 25 mL concentrated HNO_3 to 475 mL reagent water in a 500 mL wash bottle. Cap, point the dispensing tip into a sink, and shake gently to mix.

5.6 Multi-element spiking solutions (as listed in Figure 3).

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

Samples to be analyzed for dissolved metals should be filtered through a 0.45 um membrane filter and preserved as soon as possible after collection. Samples to be analyzed for total metals should be preserved, unfiltered, as soon as possible after collection. Aqueous samples are preserved by acidification with nitric acid to a pH of <2.

TITLE: **ACID DIGESTION OF AQUEOUS SAMPLES BY EPA METHOD 3010 FOR ICP AND ICP-MS ANALYSIS OF TOTAL OR DISSOLVED METALS**

Please refer to Katahdin Analytical Services SOP CA-108, “Basic Laboratory Technique”, current revision, for information on subsampling.

7.0 PROCEDURES

7.1 Prior to performing the digestion, make a list of the samples that are to be digested. Enter digestion information (Katahdin Sample Numbers, QC Batch ID, preparation date, analyst initials, etc.) into the ACCESS computer spreadsheet. Print out a copy of the spreadsheet. With a permamament marker, make sample labels and attach to the polyethylene sample containers that will contain the digestates.

7.2 If using glass beakers as the digestion vessels, submerge previously cleaned beakers three times into a 10% nitric acid bath, then rinse three times with reagent water. The polyethylene digestion tubes used in conjunction with the block digester do not require acid rinsing or precleaning. Label the digestion vessels with sample numbers.

7.3 If digestion is performed using a block digester, the sample aliquot may be measured in the digestion vessel using the graduations on the digestion tubes. Measure 50 ml of well-mixed sample into a 70 ml block digestion tube. A larger sample aliquot may be used (up to 250 mL) if concentration of the sample during digestion is desired. Sample volumes larger than 50 mL may be digested in 250 mL beakers. Measure aliquot of well-mixed sample into a graduated specimen cup and transfer into a properly cleaned 250 mL beaker. Sample volumes of more than 50ml may not be digested using the 70ml block digester tubes. The volumes of reagents and spiking solutions used must be adjusted in proportion to the final digestate volume. The reagent and spiking solution volumes listed below are based on a final volume of 50 mL.

7.4 Add spike solutions to matrix spike samples and laboratory control samples (refer to Figure 3 for spiking instructions).

7.5 Use a repipetter to add 1.5 mL of concentrated HNO_3 (per 50 mL final volume) to the sample. Cover with a ribbed watch glass and place on heatsource. Heat cautiously, without boiling the sample, and evaporate to a low volume (10 - 15 mL).

NOTE: Do not allow any portion of the bottom of the digestion vessel to go dry during any part of the digestion. If a sample is allowed to go to dryness, low recoveries may result. Should this occur, discard the digestate and re-prepare the sample.

7.6 Cool the sample and add another 1.5 mL aliquot (per 50 mL final volume) of concentrated HNO_3. Cover and resume heating, increasing the temperature until a gentle reflux action occurs.

7.7 Continue heating, adding additional acid as necessary, until the digestate is light in color or does not change in appearance with continued refluxing.

7.8 Evaporate digestate to a low volume (10 - 15 mL).

7.9 Cool the sample and use a repipetter to add 5 mL (per 50 mL final volume) of 1:1 HCl. Cover the sample and resume heating, refluxing for an additional 15 minutes to dissolve any precipitate or residue resulting from evaporation.

7.10 Allow the sample to cool.

7.11 If the digestate contains visible particulate material, it must be filtered. Use a pre-cleaned funnel and acid-rinsed filter paper to filter the digestate into a clean graduated plastic specimen container or block digester digestion tube. Using a wash bottle, rinse the digestion vessel with reagent water and add the rinsates to the filter apparatus. After all of the liquid in the filter has drained into the specimen container or digestion tube, thoroughly rinse the filter three times with small (5-10 mL) volumes of reagent water, allowing the liquid to drain completely after each rinse.

If the digestion was performed using hot plates and the digestate does not contain particulate material, simply decant the digestate into a clean graduated specimen container (or graduated sample container with attached snap lid), rinse the beaker with reagent water, and add the rinsates to the container.

If the digestion was performed using a block digester and the digestate contains no visible particulate material, the digestate may be brought to final volume and stored in the digestion tube without decanting or rinsing.

7.12 Using the graduations on the specimen container, snap-lid container or digestion tube, dilute to the required final volume with reagent water. If a specimen container has been used, transfer the contents to the corresponding labeled polyethylene sample bottle, cap the bottle, and discard the empty specimen container. If a snap-lid container or digestion tube has been used, close and secure the snap-lid. Shake the container gently to mix. The digestate is now ready for analysis.

7.13 Review the ACCESS computer spreadsheet for accuracy. If any information is incorrect, make the necessary changes to the computer spreadsheet and print out a corrected copy. Do not discard the original copy of the spreadsheet. Record (hand write) the sample bottle ID, reagent lot numbers, spiking information, initial and final volumes, hot plate ID and hot plate temperature in the appropriate spaces on the spreadsheet. Record any method deviations, irregularities with the samples, or other pertinent observations at the bottom of the page, and sign and date the spreadsheet. Bind all copies of the spreadsheet in the sample preparation log. An example sample preparation logbook page (ACCESS spreadsheet) is included as Figure 1.

7.14 Place each batch of digestates in a box labeled with the QC Batch ID, and put the box of digestates in the metals digestates storage area.

7.15 A condensation of the procedure described above is included in this SOP as Table 3. A controlled copy of this table may be posted in the metals preparation laboratory for reference by the analyst.

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

8.1 At least one preparation blank for waters (PBW) is processed concurrently with each digestion batch of 20 or fewer samples, and is used to assess contamination resulting from the digestion procedure. The PBW consists of an aliquot of reagent water that is digested using the same reagents as those used to digest associated samples. The initial and final volumes of the PBW must be identical to those of the associated samples (i.e., if the associated samples were concentrated during digestion, the PBW must also be concentrated). Refer to the appropriate analytical SOP for PBW acceptance criteria and corrective actions.

8.2 At least one laboratory control sample for waters (LCSW) is processed concurrently with each digestion batch of 20 or fewer samples. The LCSW consists of an aliquot of reagent water that is spiked to contain all analytes of interest at known concentrations, and is digested using the same reagents as those used to digest associated samples. The initial and final volumes of the LCSW must be identical to those of the associated samples (i.e., if the associated samples were concentrated during digestion, the LCSW must also be concentrated). Directions for spiking the LCSW are contained in Figures 3 and 4. The measured analyte recoveries for the LCSW are used to assess digestion method performance. Refer to the appropriate analytical SOP for LCSW recovery acceptance criteria and corrective actions.

8.3 Matrix spiked samples are processed concurrently with each digestion batch at a minimum frequency of one per digestion batch. A matrix spike sample consists of an aliquot of a sample that is spiked with known amounts of all analytes of interest. Matrix spike recoveries are used to assess the effects of sample matrix on digestion and analysis performance. Directions for spiking matrix spike samples are contained in Figures 3 and 4. Refer to the appropriate analytical SOP for matrix spike recovery acceptance criteria and corrective actions.

8.4 Matrix spiked duplicate samples are processed concurrently with each digestion batch at a minimum frequency of one per digestion batch. Matrix spiked duplicate samples are used to assess the precision of the digestion and analysis methods. Refer to the appropriate analytical SOP for matrix spike duplicate precision acceptance criteria and corrective actions.

NOTE: Clients may choose specific samples for matrix spike and matrix spike duplicate analysis; otherwise, the choice is left to the person performing the digestion. The sample volumes available may restrict the choice of samples used for matrix spike and duplicate digestion. Field blank samples should not be chosen for matrix spike and matrix spike duplicate analysis.

8.5 The quality control measures and frequencies described above are minimum requirements. They are summarized for reference in Table 1. Individual clients and analytical programs may impose additional QC requirements.

9.0 METHOD PERFORMANCE

Refer to the applicable analytical SOPs for method performance information.

10.0 APPLICABLE DOCUMENTS/REFERENCES

Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, USEPA SW846, 3rd Edition, Final Updates I, II, IIA, IIB, III, IIIA, IIIB and IV, February 2007, Method 3010A.

Department of Defense Quality Systems Manual for Environmental Laboratories (DOD QSM), Version 4.1, 04/22/09.

The National Environmental Laboratory Accreditation Conference (NELAC) Standards, June 2003.

Katahdin SOP CA-101, Equipment Maintenance and Troubleshooting, current revision.

Katahdin SOP QA-806, Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications, current revision.

LIST OF TABLES AND FIGURES

TITLE: **ACID DIGESTION OF AQUEOUS SAMPLES BY EPA METHOD 3010 FOR ICP AND ICP-MS ANALYSIS OF TOTAL OR DISSOLVED METALS**

TABLE 1

QC REQUIREMENTS

Analytical Method	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
3010	Preparation Blank for Waters (PBW)	One per prep batch of 20 or fewer samples	Refer to analytical method	Refer to analytical method
	Laboratory Control Sample for Waters (LCSW)	One per prep batch of 20 or fewer samples	Refer to analytical method	Refer to analytical method
	Matrix Spike Sample	One per prep batch	Refer to analytical method	Refer to analytical method
	Matrix Spike Duplicate Sample	One per prep batch	Refer to analytical method	Refer to analytical method
	Demonstration of analyst proficiency; accuracy and precision	One time demonstration by each analyst performing the method	Must pass all applicable QC for method	Repeat analysis until able to perform passing QC; document successful performance in personal training file

TITLE: **ACID DIGESTION OF AQUEOUS SAMPLES BY EPA METHOD 3010 FOR ICP AND ICP-MS ANALYSIS OF TOTAL OR DISSOLVED METALS**

TABLE 2

SUMMARY OF METHOD MODIFICATIONS

TOPIC	KATAHDIN SOP CA-604-05	EPA METHOD 3010, current revision
Apparatus/Materials	1) Disposable plastic specimen cup used to measure sample volume. 2) Digestion performed in 250 mL, 400 mL Griffin beaker, or 70ml digestion tube to facilitate evaporation. 3) Ribbed watch glass used throughout digestion to reduce contamination.	1) Graduated cylinder used to measure sample volume. 2) Digestion performed in 150 mL Griffin beaker. 3) Ribbed and non-ribbed watch glasses alternated in digestion.
Procedures	1) Digestate may be analyzed for antimony and silver. 2) Sample aliquots larger or smaller than 100 mL may be used. 3) Sample evaporated to 10 - 15 mL.	1) Digestate may not be analyzed for antimony and silver. 2) Requires sample aliquot of 100 mL. 3) Sample evaporated to 5 mL.

TITLE: **ACID DIGESTION OF AQUEOUS SAMPLES BY EPA METHOD 3010 FOR ICP AND ICP-MS ANALYSIS OF TOTAL OR DISSOLVED METALS**

TABLE 3

PROCEDURE CONDENSATION: EPA METHOD 3010

1. If performing digestion on a hot plate, rinse glass beakers and ribbed watch glasses 3 times in acid bath. Then rinse beakers and watch glasses 3 times with reagent water. If performing digestion with block digester, polyethylene digestion tubes do not require precleaning.
2. Label digestion vessels with sample numbers.
3. Mix sample well, measure 50 mL (or smaller or larger aliquot) into a polyethylene digestion tube. If using glass beakers, measure aliquot into graduated specimen container, and transfer to appropriate digestion vessel.
4. Add spike solutions to matrix spike samples and LCSW (refer to Figure 3 of this SOP).
5. Add 1.5 mL (per 50 mL final volume) concentrated HNO_3 to sample.
6. Cover with a ribbed watch glass.
7. Place on heating device (hotplate or block digester) and evaporate to 10 - 15 mL.
8. Cool sample and add another 1.5 mL (per 50 mL final volume) concentrated HNO_3.
9. Resume heating until gentle reflux action occurs.
10. Continue heating, adding additional HNO_3 as necessary until digestion is complete.
11. Evaporate to 10 - 15 mL.
12. Cool sample and add 5 mL (per 50 mL final volume) 1:1 HCl. Resume heating and reflux gently for 15 minutes.
13. Cool sample and filter (if necessary) or decant into a graduated polyetheyne digestion tube. Rinse beaker with reagent water and filter or decant rinsate into specimen container.
14. Dilute to appropriate final volume with reagent water.
15. Cap sample container and shake gently to mix.

FIGURE 1

TITLE: **ACID DIGESTION OF AQUEOUS SAMPLES BY EPA METHOD 3010 FOR ICP AND ICP-MS ANALYSIS OF TOTAL OR DISSOLVED METALS**

EXAMPLE PAGE FROM METALS SAMPLE PREPARATION LOGBOOK

Katahdin Analytical Services, Inc. — **Metals Preparation Benchsheet**

Reagent Information:
JT Baker HNO3: H14024 JT Baker HCL: IT3907 KMG H2O2: N/A **Method:** 3010

LCS / Spike **LCS/Spiking Information:**

☑	☑	LV. CLPP-SPK-1 (ID/Vol):	MS1404 / 0.05	mL	Hot Plate/Block ID : A
☑	☑	CLPP-SPK-INT1 (ID/Vol):	MW2021 / 0.5	mL	Start Time/Temp.: 930 / 95 °C
☑	☑	CLPP-SPK-INT2 (ID/Vol):	MW2209 / 0.5	mL	End Time/Temp. : 1500 / 95 °C
☑	☑	Uranium Spike (ID/Vol):	MS1594 / 0.005	mL	Thermometer ID/Pos.: ALC 8 / 1.4
☐	☐	CLPP-SPK-4 (ID/Vol):	N/A /	mL	

Fisher Filter Paper: 411672325 2

Sample ID	Batch ID	Initial Wt/Vol	Initial Units	Final Vol	Final Units	MX	Meth	Anal.	Date	Initial Color	Initial Clarity	Final Color	Final Clarity	Artifacts	Bottle
LCSWAB011CW0	AB011CW0	0.05	L	0.05	L	AQ	IC	AJB	02/01/2010	N/A	N/A	N/A	N/A		
PBWAB011CW0	AB011CW0		L		L	AQ	IC	AJB	02/01/2010	N/A	N/A	N/A	N/A		
SD0405-001	AB011CW0		L		L	AQ	IC	AJB	02/01/2010						A
SD0405-001P	AB011CW0		L		L	AQ	IC	AJB	02/01/2010						
SD0405-001S	AB011CW0		L		L	AQ	IC	AJB	02/01/2010						
SD0405-002	AB011CW0		L		L	AQ	IC	AJB	02/01/2010						
SD0405-003	AB011CW0		L		L	AQ	IC	AJB	02/01/2010						
SD0405-004	AB011CW0		L		L	AQ	IC	AJB	02/01/2010						
SD0405-005	AB011CW0		L		L	AQ	IC	AJB	02/01/2010						
SD0422-001	AB011CW0		L		L	AQ	IC	AJB	02/01/2010						B
SD0423-001	AB011CW0		L		L	AQ	IC	AJB	02/01/2010						A
SD0429-001	AB011CW0		L		L	AQ	IC	AJB	02/01/2010						B
SD0429-002	AB011CW0		L		L	AQ	IC	AJB	02/01/2010						
SD0455-001	AB011CW0		L		L	AQ	IC	AJB	02/01/2010						
SD0455-002	AB011CW0		L		L	AQ	IC	AJB	02/01/2010						
SD0455-003	AB011CW0		L		L	AQ	IC	AJB	02/01/2010						
SD0455-004	AB011CW0		L		L	AQ	IC	AJB	02/01/2010						

AJB 2-1-10

Digestion performed by: AJB On: 2-1-10

Page: AB003 **Revision:** 00

TITLE: **ACID DIGESTION OF AQUEOUS SAMPLES BY EPA METHOD 3010 FOR ICP AND ICP-MS ANALYSIS OF TOTAL OR DISSOLVED METALS**

FIGURE 2

PREPARATION OF MATRIX SPIKES, LABORATORY CONTROL SAMPLES, AND SPIKING SOLUTIONS FOR DIGESTION OF AQUEOUS SAMPLES BY METHOD 3010

Sample or Solution Name	Component Solution Name	Source of Component	Amount of Component Added per 50 mL Final Volume (mL)
Laboratory Control Sample (LCSW) and Matrix Spike	CLPP-SPK-1	Inorganic Ventures	0.050
	CLPP-SPK-INT1	Lab Prepared (see below)	0.50
	CLPP-SPK-INT2	Lab Prepared (see below)	0.50
	1000 mg/L Uranium Standard	Inorganic Ventures	0.005

Sample or Solution Name	Component Solution Name	Source of Component	Amount of Component Added per 100 mL Final Volume (mL)
CLPP-SPK-INT1	1000 mg/L Se	High Purity Standards	1.0
	1000 mg/L As	High Purity Standards	1.0
	1000 mg/L Pb	High Purity Standards	1.0
	1000 mg/L Cd	High Purity Standards	2.5
	1000 mg/L Sb	High Purity Standards	1.0
	10000 mg/L K	High Purity Standards	10.0
	10000 mg/L Na	High Purity Standards	7.5
	10000 mg/L Mg	High Purity Standards	5.0
	10000 mg/L Ca	High Purity Standards	2.5
CLPP-SPK-INT2	2007ICS-1	Inorganic Ventures	10.0
	1000 mg/L Sr	High Purity Standards	5.0
	1000 mg/L Sn	High Purity Standards	5.0
	10000 mg/L Si	High Purity Standards	5.0

TITLE: **ACID DIGESTION OF AQUEOUS SAMPLES BY EPA METHOD 3010 FOR ICP AND ICP-MS ANALYSIS OF TOTAL OR DISSOLVED METALS**

FIGURE 3

ELEMENT CONCENTRATIONS IN MATRIX SPIKES, LABORATORY CONTROL SAMPLES, AND THEIR COMPONENT SPIKING SOLUTIONS FOR DIGESTION OF AQUEOUS SAMPLES BY METHOD 3010

Element	CONCENTRATION IN SOLUTION, mg/L							
	Matrix Spike	LCSW	CLPP-SPK-1	CLPP-SPK-4	CLPP-SPK-INT1	CLPP-SPK-INT2	2007 ICS-1	1000 mg/L U
Aluminum	2.000	2.000	2000					
Antimony	0.500	0.500		100	100			
Arsenic	0.500	0.500		4	10			
Barium	2.000	2.000	2000					
Beryllium	0.050	0.050	50					
Boron	0.500	0.500		50		50	500	
Cadmium	0.250	0.250		5	25			
Calcium	2.500	2.500			250			
Chromium	0.200	0.200	200					
Cobalt	0.500	0.500	500					
Copper	0.250	0.250	250					
Iron	1.000	1.000	1000					
Lead	0.500	0.500		2	10			
Magnesium	5.000	5.000			500			
Manganese	0.500	0.500	500					
Molybdenum	0.300	0.300		30		30	300	
Nickel	0.500	0.500	500					
Potassium	10.000	10.000			1000			
Selenium	0.500	0.500		5	50			
Silicon	5.230	5.230				523	230	
Silver	0.050	0.050	50					
Sodium	7.500	7.500			750			
Strontium	0.500	0.500		50		50		
Thallium	0.500	0.500		5	10			
Tin	0.500	0.500		50		50		
Titanium	1.000	1.000		100		100	1000	
Uranium	0.100	0.100						1000
Vanadium	0.500	0.500	500					
Zinc	0.500	0.500	500					

TITLE: ACID DIGESTION OF SOLID SAMPLES BY USEPA METHOD 3050 FOR METALS ANALYSIS BY ICP-AES AND GFAA

Prepared By: George Brewer Date: 3/98

Approved By:

Group Supervisor: George Brewer Date: 01/24/01

Operations Manager: Jol C. Burton Date: 1/24/01

QA Officer: Deborah J. Nadeau Date: 1.24.01

General Manager: Duncan F. Kinsman Date: 1/25/01

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
01 3050B	Format changes, added pollution prevention, added MSD, added spiking instruction tables.	DN	1.24.01	1/24/01
02 3050B	Removed all references/procedures devoted to GFAA. Added use of digestates for ICP/MS analysis. Revised standard solution names & concs. in Tables 3 & 4 to reflect current practice.	DN	8.29.02	8.29.02
03 3050B	New Title to include ICMOS, 3. Use of digestion block and polyethylene digestion tubes added to sections 4.0, 7.0 and Table 1. PBS changed from 1.0g water to 1.0g boiling chips. H_2O_2 addition from 3.0ml then 7.0mls to 2.0ml, 2.0ml then 7.0ml. Figures and Tables updated to reflect current practices.	LAD	03/08	03/08
04	Updated Tables 3 and 4 with current spike concentrations and volumes added. Updated logbook page. Added CA-108 reference for subsampling information.	LAD	08/09	08/09

TITLE: ACID DIGESTION OF SOLID SAMPLES BY USEPA METHOD 3050 FOR METALS ANALYSIS BY ICP-AES, ICP-MS

Please acknowledge receipt of this standard operating procedure by signing and dating both of the spaces provided. Return the bottom half of this sheet to the QA Department.

I acknowledge receipt of copy ___ of document **SOP CA-605-04**, titled **ACID DIGESTION OF SOLID SAMPLES BY USEPA METHOD 3050 FOR METALS ANALYSIS BY ICP-AES, ICP-MS AND ILM05.3.**

Recipient: _____ Date: _____

KATAHDIN ANALYTICAL SERVICES, INC.
STANDARD OPERATING PROCEDURE

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Recipient: _____ Date: _____

TITLE: ACID DIGESTION OF SOLID SAMPLES BY USEPA METHOD 3050 FOR METALS ANALYSIS BY ICP-AES, ICP-MS

1.0 SCOPE AND APPLICATION

The purpose of this SOP is to describe the Katahdin Analytical Services, Inc. procedure utilized to dissolve solid matrices and solubilize metals from solid samples prior to analysis for metals by ICP-AES and ICP-MS. This SOP applies to samples prepared by EPA Method 3050, with method modifications as summarized in Table 2.

This procedure applies to all solid sample (e.g. sediments, sludges, soils, and ashes) preparations for ICP-AES and ICP-MS analyses. This method is not a total ___ digestion technique for most samples. It is a very strong acid digestion that will dissolve almost all elements that could become "environmentally available". By design, elements bound in silicate structures are not normally dissolved by this procedure as they are not usually mobile in the environment.

1.1 Definitions

ICP-AES – Inductively Coupled Plasma Atomic Emission Spectroscopy.

ICP-MS – Inductively Coupled Plasma Mass Spectrometry.

LCSS – Laboratory Control Sample for Solids – A standard or solid reference material that has been brought through the sample preparation process.

Matrix Spike – An aliquot of a sample to which a known amount of analyte has been added before digestion.

PBS – Preparation Blank for Solids – An aliquot of reagent water that has been brought through the sample preparation process.

1.2 Responsibilities

This method is restricted to use by, or under the supervision of analysts experienced in the acid digestion of solid samples by USEPA Method 3050 for metals analysis. Each analyst must demonstrate the ability to generate acceptable results with this method. Refer to Katahdin SOP QA-805, current revision, "Personnel Training & Documentation of Training".

It is the responsibility of all Katahdin technical personnel involved in the acid digestion of solid samples by USEPA Method 3050 to read and understand this SOP, to adhere to the procedures outlined, and to properly document their data in the appropriate lab notebook. Any deviations from the procedure or irregularities with the samples should also be recorded in the lab notebook and reported to the responsible Department Manager or designated qualified data reviewer.

It is the responsibility of the Department Manager to ensure that technical personnel perform acid digestions in accordance with this SOP and to confirm that their work is properly documented through periodic review of the associated logbooks.

1.3 Safety

The acids used in this procedure are highly corrosive and reactive, and spiking standards contain toxic metals. The toxicity and reactivity of client samples are usually unknown, so samples should always be assumed to present a contact hazard. To reduce or eliminate exposure to potentially harmful chemicals, lab coats, gloves, and safety glasses or goggles must be worn whenever handling samples or reagents. Additional safety apparel, including face shields, aprons, dust masks, and shoe protectors, is available in the Metals prep lab and should be worn whenever circumstances warrant.

Acids should be added to samples slowly and carefully, while watching for reactions. This should be done under a hood, in case harmful fumes are evolved.

Hood sashes should be lowered as far as possible whenever beakers are being heated on a hot plate. Use caution when handling hot beakers.

Personnel are required to read the Katahdin Hazrdous Waste Management Plan and Safety Manual before performing this procedure, and must be familiar with the general rules for laboratory safety, personal hygiene, housekeeping, and use of protective clothing and equipment.

1.4 Pollution Prevention/Waste Disposal

Whenever possible, laboratory personnel should use pollution prevention techniques to address their waste generation. Refer to the current revision of the Katahdin Hazardous Waste Management Program for further details on pollution prevention techniques.

Excess spiking solutions must be emptied into the corrosive waste carboy located in the Metals prep lab for subsequent appropriate disposal in accordance with the Katahdin Hazardous Waste Management Plan and Safety Manual.

Sample digestates should be stored for a minimum of 60 days after digestion to allow for analysis, and reanalysis if necessary. Digestates older than 60 days may be emptied into the corrosive waste carboy in the Metals prep lab for subsequent appropriate disposal in accordance with the Katahdin Hazardous Waste Mnagement Plan and Safety Manual.

TITLE: ACID DIGESTION OF SOLID SAMPLES BY USEPA METHOD 3050 FOR METALS ANALYSIS BY ICP-AES, ICP-MS

2.0 SUMMARY OF METHOD

A representative 1 to 2 g (wet weight) sample is digested with repeated additions of nitric acid and hydrogen peroxide. Hydrochloric acid is added to the initial digestate and the sample is refluxed. The digestate is then filtered and diluted to a final volume of 100 mL.

3.0 INTERFERENCES

Interferences are discussed in the applicable analytical SOPs.

4.0 APPARATUS AND MATERIALS

4.1 Digestion vessels. If digestion is performed using a hot plate, the appropriate digestion vessels are 100 mL pre-cleaned Griffin beakers (cleaned according to the current revision of SOP CA-100, "Labware Cleaning" and CA-602, “Glassware Preparation and Sample Preservation for Trace Element Analyses”). If digestion is performed using a block digester, the appropriate digestion vessels are new 70 mL disposable graduated polyethylene digestion tubes with attached snap lids.

4.2 Ribbed watch glasses. If digestion is performed using a hot plate, 75 mm diameter glass watch glasses (pre-cleaned as above) are used. If digestion is performed using a block digester, 40 mm diameter disposable polyethylene watch glasses are used.

4.3 Adjustable volume automatic pipets covering the range from 10 uL to 1000 uL and disposable pipet tips; calibrated Finn pipets or Eppendorf pipets are acceptable.

4.4 Disposable graduated polystyrene specimen containers with pouring lips, 200 mL capacity.

4.5 Hot plate or block digester, griddle, or other heating source - adjustable and capable of maintaining a temperature of 95 ^{o}C ± 5 ^{o}C. Heating sources must be numbered for easy identification.

4.6 Device for measuring hot plate temperature, consisting of a flask or digestion vessel in which the bulb of a thermometer is immersed in sand or water. The temperature of each hot plate used is measured and recordedeach day. The hot plate identification number and the measured temperature are recorded on the sample preparation logbook sheet.

4.7 Plastic funnels, pre-cleaned as in Section 4.1.

4.8 Filter funnel holders, capable of suspending plastic funnels above disposable specimen containers.

4.9 Polyethylene wash bottles for dispensing reagent water and 5% HNO_3.

4.10 Filter paper, Whatman No. 41 or equivalent. Filters are acid-washed immediately prior to use as follows. Place a pre-cleaned funnel in the funnel holder and put a disposable plastic specimen container under the funnel to collect the rinsates. Place a folded filter in the funnel and rinse three times with approximate 10 mL volumes of 5% HNO_3, making sure the entire surface of the filter is wetted each time and allowing each rinse to drain completely before continuing. Then rinse three times with approximate 25 mL volumes of reagent water, again allowing each rinse to drain completely. Discard the rinsates into the appropriate waste container. The acid-washed filter is now ready for use.

4.11 Polyethylene sample containers with screw caps or graduated polyethylene sample containers with attached snap lids, 125 mL capacity.

4.12 Repipetters (adjustable repeating pipetters with reservoirs) for dispensing concentrated nitric acid, 1:1 HNO_3, and concentrated HCl.

4.13 Analytical balance capable of reading to 0.01 gram.

4.14 Spatulas, scoops, or spoons; plastic or stainless steel, rinsed with 5% HNO_3 and reagent water. Disposable tongue depressors may be used and do not requrire to be rinsed.

5.0 REAGENTS

5.1 Concentrated nitric acid, HNO_3 – trace metals grade.

5.2 Concentrated hydrochloric acid, HCl – trace metals grade.

5.3 Reagent water - water that meets the performance specifications of ASTM Type II water (ASTM D1193).

5.4 Nitric acid, 1:1. Add a volume of concentrated HNO_3 to an equivalent volume of reagent water and swirl gently to mix.

5.5 Nitric acid, 5% v/v. Add 25 mL concentrated HNO_3 to 475 mL reagent water in a 500 mL wash bottle. Cap, point the dispensing tip into a sink, and shake gently to mix.

5.6 30% hydrogen peroxide (H_2O_2) - spectrometric grade.

5.7 Multielement spiking solutions (see Table 3 for a list of required spiking solutions).

5.8 Solid reference material – a soil containing all the elements of interest, with empirically established method-specific recoveries and acceptance limits for all analytes. Solid reference materials are purchased with documentation of analysis provided by the vendor. See Figure 4 for an example certificate of analysis for a solid reference material.

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

Samples should be collected in clean plastic or glass containers. Samples must be refrigerated (4°C ±2°C) upon receipt by the laboratory. The holding time for solid samples is 6 months from the date of sample collection.

7.0 PROCEDURE

The procedure described below is condensed for quick reference in Table 3.

SAMPLE PREPARATION

7.1 Prior to performing the digestion, make a list of the samples that are to be digested. Enter digestion information (Katahdin Sample Numbers, QC Batch ID, preparation date, analyst initials, etc.) into the ACCESS computer spreadsheet. Print out a copy of the spreadsheet (see Figure 2 for an example). Hand label the digestate vessels

7.2 If using glass beakers as the digestion vessels, submerge previously cleaned beakers and watch glasses three times into a 10% nitric acid bath, then rinse three times with reagent water. The polyethylene digestion tubes used in conjunction with the block digeter do not require acid rinsing or precleaning. Label the digestion vessels with sample numbers.

7.3 Weigh 1 to 2 g of well-mixed sample into a properly cleaned, labeled, and tared Griffin beaker or polyethylene digestion tube. Record (hand write) the weight of each sample on the printout of the digestion spreadsheet. Refer to Katahdin Analytical Services SOP CA-108, current revision “Basic Laboratory Technique” for more information on subsampling.

7.4 Weigh an appropriate amount of solid reference material to a clean, labeled, and tared Griffin beaker or polyethylene digestion tube to serve as a laboratory control sample.

7.5 Add spike solutions to matrix spike samples (refer to Tables 3 and 4 for spiking instructions).

7.6 Using repipetters, add 10 mL of 1:1 HNO_3, mix the slurry. Cover with a ribbed watch glass and place on heat source. Gently heat the sample to 95 ^{o}C ± 5 ^{o}C and reflux for 10 to 15 minutes without boiling. Remove the digestion vessel from the heat source and cool the sample.

7.7 Add 5 mL of concentrated HNO_3 to the sample, replace the watch glass, and reflux for 30 minutes. If brown fumes are generated, indicating oxidation of the sample by HNO_3, repeat this step (addition of 5 mL of concentrated HNO_3) until no brown fumes are given off by the sample, indicating complete reaction by HNO_3.

7.8 Continue heating the sample at 95 ^{o}C ± 5^{o}C without boiling until the digestate has evaporated to approximately 5 to 10 mL or until two hours have elapsed, whichever occurs first. Do not allow the sample to go to dryness. Remove the digestion vessel from the heat source and cool the sample.

7.9 Add 2 mL of reagent water and 2 mL of 30% H_2O_2 to the sample, replace the watch glass, and heat gently on the heat source to start the peroxide reaction. Continue heating until effervescence subsides.

7.10 Add an additional 2 mL of 30% H_2O_2 to the sample, replace the watch glass, and heat gently on the heat source to start the peroxide reaction. Continue heating until effervescence subsides.

7.11 Add an additional 6 mL of 30% H_2O_2 in 1-mL aliquots with warming until the effervescence is minimal or until the general sample appearance is unchanged.

7.12 Continue heating the sample at 95 ^{o}C ± 5^{o}C without boiling until the digestate has evaporated to approximately 5 to 10 mL or until two hours have elapsed, whichever occurs first. Do not allow the sample to go to dryness. Remove the sample from the heat source and cool.

7.13 Add 10 mL of concentrated HCl to the digest from 7.12, replace the watch glass, and reflux at 95^{o}C ± 5^{o}C for 15 minutes. Remove the sample from the heat source and cool.

7.14 Use a pre-cleaned funnel and acid-rinsed filter paper to filter the digestate into a clean graduated polystyrene specimen container or graduated polyethylene sample container with attached snap lid. Using a wash bottle, rinse the digestion vessel with reagent water and add the rinsates to the filter apparatus. After all of the liquid in the filter has drained into the specimen container, thoroughly rinse the filter three times with small (5-10 mL) volumes of reagent water, allowing the liquid to drain completely after each rinse. Using the graduations on the specimen container or snap-lid container, dilute to 100 mL with reagent water. If a specimen container has been used, transfer the contents to the corresponding labeled polyethylene sample bottle, cap the bottle, and discard the empty specimen container. If a snap-lid

container has been used, close and secure the snap-lid. Shake the container gently to mix. The digestate is now ready for ICP-AES or ICP-MS analysis.

7.15 Review the ACCESS computer spreadsheet for accuracy. If any information is incorrect, make the necessary changes to the computer spreadsheet and print out a corrected copy. Do not discard the original copy of the spreadsheet. Record (hand write) reagent lot numbers, spiking information, and heat source temperature in the appropriate spaces on the spreadsheet. Record any method deviations, irregularities with the samples, or other pertinent observations at the bottom of the page, and sign and date the spreadsheet. Bind all copies of the spreadsheet in the sample preparation log. An example sample preparation logbook page (ACCESS spreadsheet) is included as Figure 2.

7.15 Reopen the electronic ACCESS spreadsheet for the digestion and transcribe the sample weights from the handwritten, bound copy into the electronic copy. The information in this electronic spreadsheet will later be imported into the ACCESS metals database and used to calculate sample concentrations on a weight basis.

7.16 Place each batch of digestates in a box labeled with the QC Batch ID, and put the box of digestates in the metals digestates storage area.

CALCULATIONS

7.17 Analytical results for solid samples are reported on a dry weight basis. Total solids are determined by the Wet Chemistry Group, and are recorded in spreadsheets that are electronically imported into the Access metals database. Final dry weight concentrations are calculated by the Access database as follows:

$$\text{Concentration (mg/kg dry weight)} = (C \times V) / (W \times S)$$

where: C = Measured concentration (mg/L)
V = Digestate final volume (L)
W = Sample wet weight (kg)
S = % Solids/100

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

USEPA Method 3050 requires the laboratory to perform specific quality control checks to assess laboratory performance and data quality. Minimum frequencies, acceptance criteria, and corrective actions for these control checks are tabulated in Table 1 and are described below. Table 1 criteria are intended to be guidelines for analysts. The table does not cover all possible situations. If any of the QC requirements are outside the recovery ranges listed in Table 1, all associated samples must be evaluated against all the QC. In some cases data may be reported, but may be reanalyzed in other cases. Making new reagents and

standards may be necessary if the standardization is suspect. The corrective actions listed in Table 1 may rely on analyst experience to make sound scientific judgements. These decisions are based on holding time considerations and client and project specific Data Quality Objectives. The Depatment Manager, Operations Manager, and/or Quality Assurance Officer may be consulted to evaluate data. Some samples may not be able to be reanalyzed within hold time. In these cases "qualified" data with narration may be advisable after consultation with the client.

8.1 At least one preparation blank for soils (PBS) is processed concurrently with each digestion batch of 20 or fewer samples, and is used to assess contamination resulting from the digestion procedure. The PBS consists of a 1.0 g of boiling stones that is digested using the same reagents as those used to digest associated samples. Refer to the appropriate analytical SOP for PBS acceptance criteria and corrective actions.

8.2 At least one laboratory control sample for soils (LCSS) is processed concurrently with each digestion batch of 20 or fewer samples. The LCSS consists of an aliquot of a solid reference material for which the concentrations of the analytes of interest have been empirically established (solid-matrix LCSS), or an aliquot of reagent water that is spiked to contain all analytes of interest at known concentrations (aqueous-matrix LCSS). The solid reference material should normally be used as the LCSS, unless a particular client or analytical program requires that spiked reagent water be used. The LCSS is digested using the same reagents as those used to digest associated samples. Directions for spiking the aqueous-matrix LCSS are contained in Table 3. The measured analyte recoveries for the LCSS are used to assess digestion method performance. Refer to the appropriate analytical SOP for LCSS recovery acceptance criteria and corrective actions.

8.3 Matrix spike samples are processed along with each digestion batch at a minimum frequency of one per digestion batch. A matrix spike sample consists of an aliquot of a sample that is fortified with known amounts of all analytes of interest prior to digestion. Matrix spike recoveries are used to assess the biasing effects of sample matrix on digestion and analysis performance. Directions for spiking matrix spike samples are contained in Figure 2. Refer to the appropriate analytical SOP for matrix spike recovery acceptance criteria and corrective actions.

8.4 Matrix spiked duplicate samples are processed concurrently with each digestion batch at a minimum frequency of one per digestion batch. Matrix spiked duplicate samples are used to assess the precision of the digestion and analysis methods. Refer to the appropriate analytical SOP for matrix spike duplicate precision acceptance criteria and corrective actions.

NOTE: Clients may choose specific samples for matrix spike and duplicate analysis; otherwise, the choice is left to the person performing the digestion.

8.5 The quality control measures and frequencies described above are minimum requirements. Individual clients and analytical programs may impose additional QC requirements.

9.0 METHOD PERFORMANCE

Refer to the applicable instrumental analysis SOP for method performance information.

10.0 APPLICABLE DOCUMENTS/REFERENCES

"Test Methods for the Evaluation of Solid Waste," United States Environmental Protection Agency, SW-846, Third Edition, Final Update III, 12/96, Method 3050B.

Department of Defense Quality Systems Manual for Environmental Laboratories (DOD QSM), Version 4.1, 04/22/09.

The National Environmental Laboratory Accreditation Conference (NELAC) Standards, June 2003.

LIST OF TABLES AND FIGURES

TABLE 1

QC REQUIREMENTS – METHOD 3050

Method	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
3050	Preparation Blank for Solids (PBS)	One per prep batch of 20 or fewer samples.	Refer to analytical method.	Refer to analytical method.
	Laboratory Control Sample for Solids (LCSS)	One per prep batch of 20 or fewer samples.	Refer to analytical method.	Refer to analytical method.
	Matrix Spike Sample	One per prep batch.	Refer to analytical method.	Refer to analytical method.
	Matrix Spike Duplicate Sample	One per prep batch.	Refer to analytical method.	Refer to analytical method.
	Demonstration of analyst proficiency	One-time demonstration by each analyst performing the method.	Must pass all applicable QC for method.	Repeat analysis until able to perform passing QC; document successful performance in personal training file.

TITLE: ACID DIGESTION OF SOLID SAMPLES BY USEPA METHOD 3050 FOR METALS ANALYSIS BY ICP-AES, ICP-MS

TABLE 2

SUMMARY OF METHOD MODIFICATIONS – METHOD 3050

Topic	Katahdin SOP CA-605-02	Method 3050, current revision
Apparatus /Materials	1) Digestion performed in 100 mL Griffin beaker or 70 mL polyethylene tube. 2) Graduated disposable plastic cup or 120 mL polyethylene tube used to bring digestate to final volume.	1) Digestion performed in 250 mL Griffin beaker. 2) Volumetric flask used to bring digestate to final volume.
Procedure	1) Digestate volume reduced to 5 to 10 mL prior to filtering. 2) After filtration, the filters are rinsed three times with reagent water. 3) 30% H_2O_2 is added in two 2 mL aliquots and then six 1 mL aliquots.	1) Digestate volume reduced to 5 mL prior to filtering. 2) After filtration, the filters are rinsed twice with reagent water. 3) 30% H_2O_2 is added in one 3 mL aliquot and then seven 1 mL aliquots.

TABLE 3

PREPARATION OF MATRIX SPIKES AND SPIKING SOLUTIONS FOR DIGESTION OF SOLID SAMPLES BY USEPA METHOD 3050

Sample or Solution Name	Component Solution Name	Source of Component	Amount of Component Added per 100 mL Final Volume (mL)
Matrix Spike for ICP-AES	CLPP-SPK-1	Inorganic Ventures	0.10
	CLPP-SPK-INT1	Lab Prepared (see below)	1.00
	CLPP-SPK-INT2	Lab Prepared (see below)	1.00
	1000 mg/L Uranium Std.	High Purity Standards	0.01

Sample or Solution Name	Component Solution Name	Source of Component	Amount of Component Added per 100 mL Final Volume (mL)
CLPP-SPK-INT1	1000 mg/L Se	High Purity Standards	5.0
	1000 mg/L As	High Purity Standards	5.0
	1000 mg/L Pb	High Purity Standards	5.0
	1000 mg/L Cd	High Purity Standards	2.5
	1000 mg/L Sb	High Purity Standards	5.0
	1000 mg/L K	High Purity Standards	10.0
	1000 mg/L Na	High Purity Standards	7.5
	1000 mg/L Mg	High Purity Standards	5.0
	1000 mg/L Ca	High Purity Standards	2.5
CLPP-SPK-INT2	2007ICS-1	Inorganic Ventures	10.0
	1000 mg/L Sr	High Purity Standards	5.0
	1000 mg/L Sn	High Purity Standards	5.0
	10000 mg/L Si	High Purity Standards	5.0

TITLE: ACID DIGESTION OF SOLID SAMPLES BY USEPA METHOD 3050 FOR METALS ANALYSIS BY ICP-AES, ICP-MS

TABLE 4

ELEMENT CONCENTRATIONS IN ICP-AES MATRIX SPIKES AND THEIR COMPONENT SPIKING SOLUTIONS FOR DIGESTION OF SOLID SAMPLES BY METHOD 3050

Element	CONCENTRATION IN SOLUTION, mg/L							
	Matrix Spike (ICP-AES)	CLPP-SPK-1	CLPP-SPK-4	CLPP-SPK-INT1	CLPP-SPK-INT2	QCP-CICV-3 SPK-3	2007 ICS- 1	1000 mg/L U
Aluminum	2.000	2000						
Antimony	0.500		100	50				
Arsenic	0.500		4	50		500		
Barium	2.000	2000						
Beryllium	0.050	50						
Boron	0.500		50		50		500	
Cadmium	0.250		5	25		250		
Calcium	2.500			250				
Chromium	0.200	200						
Cobalt	0.500	500						
Copper	0.250	250						
Iron	1.000	1000						
Lead	0.500		2	50		500		
Magnesium	5.000			500				
Manganese	0.500	500						
Molybdenum	0.300		30		30		300	
Nickel	0.500	500						
Potassium	10.000			1000				
Selenium	0.500		5	50		500		
Silicon	5.230				523		230	
Silver	0.050	50						
Sodium	7.500			750				
Strontium	0.500		50		50			
Thallium	0.500		5	250	500			
Tin	0.500		50		50			
Titanium	1.000		100		100		1000	
Uranium	0.100							1000
Vanadium	0.500	500						
Zinc	0.500	500						

TITLE: ACID DIGESTION OF SOLID SAMPLES BY USEPA METHOD 3050 FOR METALS ANALYSIS BY ICP-AES, ICP-MS

FIGURE 1

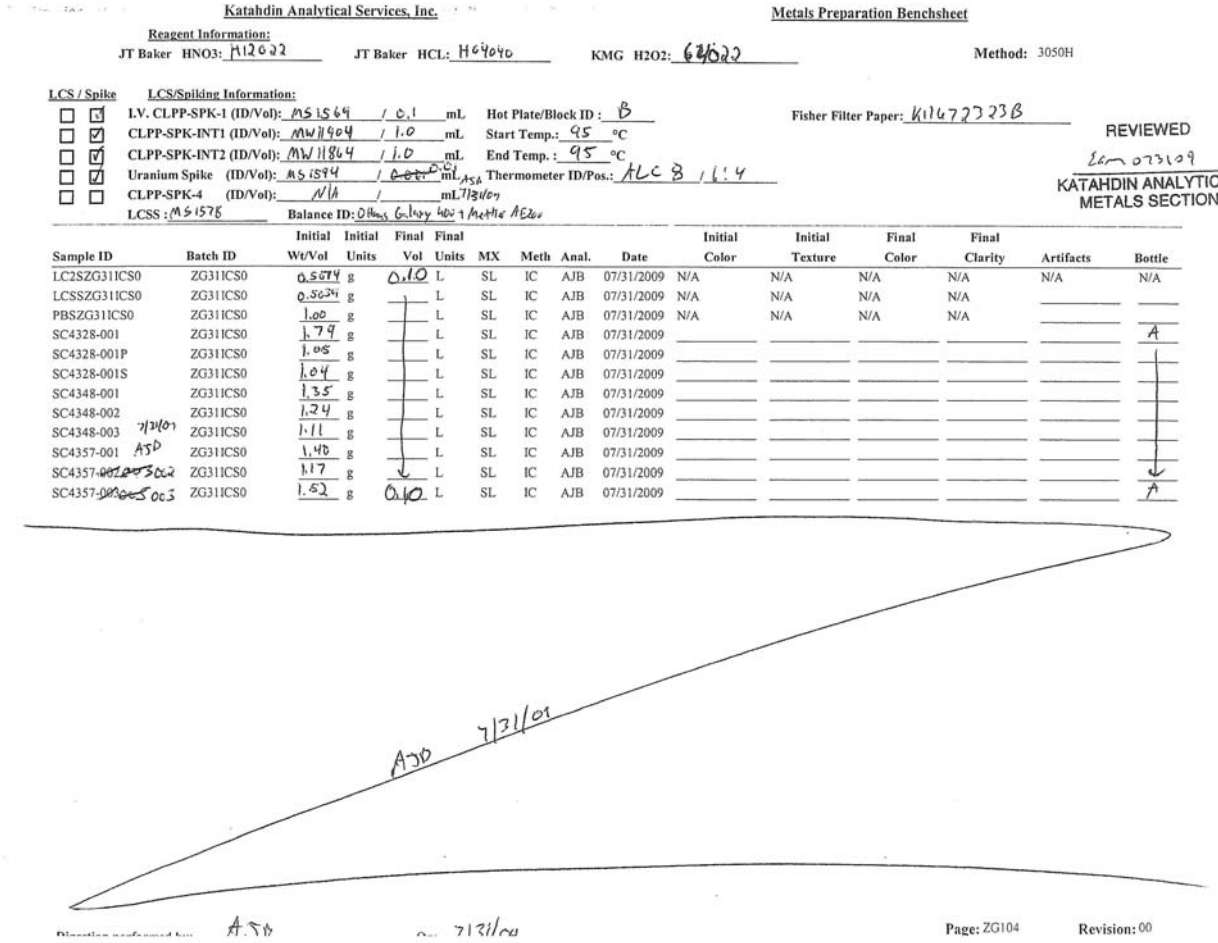
PROCEDURE CONDENSATION – METHOD 3050

1. Prepare and print out ACCESS spreadsheet.
2. If performing digestion on a hot plate, rinse 250 mL Griffin beakers and ribbed watch glasses 3 times in acid bath. Then rinse beakers and watch glasses 3 times with laboratory reagent grade water. If perfoming digestion with block digester, polyethylene digestion tubes do not require precleaning.
3. Label digestion vessels (beakers or polyethylene sample tubes) with sample numbers.
4. Weigh 1 to 2 g of well-mixed sample into tared digestion vessels. Record sample weights.
5. Add spike solutions to matrix spike samples.
6. Add 10 mL 1:1 HNO_3 to samples and cover with watch glasses.
7. Reflux for 10 to 15 minutes at $95^{o} \pm 5^{o}$ C. without boiling. Cool samples.
8. Add 5 mL conc. HNO_3, cover beakers, and reflux for 30 minutes.
9. Repeat Step 8 as necessary until digestion is complete.
10. Reduce sample volumes to 5 to 10 mL or heat for 2 hours, whichever occurs first.
11. Cool sample and add 2 mL reagent water and 2 mL 30% H_2O_2. Heat gently until effervescence subsides.
12. Cool sample and add 2 mL 30% H_2O_2. Heat gently until effervescence subsides.
13. Cool samples and add 6 mL of 30% H_2O_2 in 1 mL aliquots. Heat gently until effervescence subsides.
14. Reduce sample volumes to 5 to 10 mL or heat for 2 hours, whichever occurs first.
15. Add 10 mL conc. HCl and reflux for 10 to 15 minutes at $95^{o} \pm 5^{o}$ C.
16. Cool sample and filter into graduated specim en container. Bring to volume with reagent water and transfer to labeled polyethylene bottle.
17. Enter sample weights into ACCESS spreadsheet.

TITLE: ACID DIGESTION OF SOLID SAMPLES BY USEPA METHOD 3050 FOR METALS ANALYSIS BY ICP-AES, ICP-MS

FIGURE 2

EXAMPLE PAGE FROM METALS SAMPLE PREPARATION LOGBOOK



Katahdin Analytical Services, Inc. — Metals Preparation Benchsheet

Reagent Information: JT Baker HNO3: M12022 JT Baker HCL: H64040 KMG H2O2: 6/1/22 Method: 3050H

LCS / Spike — LCS/Spiking Information:

- LV. CLPP-SPK-1 (ID/Vol): MS1569 / 0.1 mL — Hot Plate/Block ID: B — Fisher Filter Paper: K1167223B
- CLPP-SPK-INT1 (ID/Vol): MW1904 / 1.0 mL — Start Temp.: 95 °C
- CLPP-SPK-INT2 (ID/Vol): MW1864 / 1.0 mL — End Temp.: 95 °C
- Uranium Spike (ID/Vol): MS1594 / ~~0.02~~ 0.01 mL ASA — Thermometer ID/Pos.: ALC 8 / 1.4
- CLPP-SPK-4 (ID/Vol): N/A / mL 7/31/09
- LCSS: MS1578 — Balance ID: Ohaus Galaxy 400 / Mettler AE200

REVIEWED Em 073109 KATAHDIN ANALYTICAL METALS SECTION

Sample ID	Batch ID	Initial Wt/Vol	Initial Units	Final Vol	Final Units	MX	Meth	Anal.	Date	Initial Color	Initial Texture	Final Color	Final Clarity	Artifacts	Bottle
LC2SZG311CS0	ZG311CS0	0.5074	g	0.10	L	SL	IC	AJB	07/31/2009	N/A	N/A	N/A	N/A	N/A	N/A
LCSSZG311CS0	ZG311CS0	0.5034	g		L	SL	IC	AJB	07/31/2009	N/A	N/A	N/A	N/A		
PBSZG311CS0	ZG311CS0	1.00	g		L	SL	IC	AJB	07/31/2009	N/A	N/A	N/A	N/A		
SC4328-001	ZG311CS0	1.79	g		L	SL	IC	AJB	07/31/2009						A
SC4328-001P	ZG311CS0	1.05	g		L	SL	IC	AJB	07/31/2009						
SC4328-001S	ZG311CS0	1.04	g		L	SL	IC	AJB	07/31/2009						
SC4348-001	ZG311CS0	1.35	g		L	SL	IC	AJB	07/31/2009						
SC4348-002	ZG311CS0	1.24	g		L	SL	IC	AJB	07/31/2009						
SC4348-003 7/31/09	ZG311CS0	1.11	g		L	SL	IC	AJB	07/31/2009						
SC4357-001 AJD	ZG311CS0	1.40	g		L	SL	IC	AJB	07/31/2009						
SC4357-~~002~~003	ZG311CS0	1.17	g	↓	L	SL	IC	AJB	07/31/2009						↓
SC4357-~~003~~005	ZG311CS0	1.52	g	0.10	L	SL	IC	AJB	07/31/2009						A

AJD 7/31/09

Digestion performed by: AJD Date: 7/31/09 Page: ZG104 Revision: 00

TITLE: ACID DIGESTION OF SOLID SAMPLES BY USEPA METHOD 3050 FOR METALS ANALYSIS BY ICP-AES, ICP-MS

FIGURE 3

EXAMPLE CERTIFICATE OF ANALYSIS FOR SOLID REFERENCE MATERIAL

ENVIRONMENTAL RESOURCE ASSOCIATES®
The Industry Standard™

M51475

DataPacK™
Lot No. D051-540

Trace Metals in Soil
Catalog No. 540

Certification

Method 3050 HNO3, H2O2, HCl Parameter	Total Concentration [1] (mg/Kg)	Certified Value [2] (mg/Kg)	Performance Acceptance Limits™ [3] (mg/Kg)
aluminum	55600*	7870	4630 - 11100
antimony	160	70.5	D.L. - 149
arsenic	316	289	234 - 344
barium	869	211	174 - 247
beryllium	60.9	54.4	45.2 - 63.6
boron	129	91.3	58.8 - 124
cadmium	114	101	82.9 - 119
calcium	9750*	3680	2970 - 4390
chromium	249	224	180 - 268
cobalt	113	101	82.7 - 119
copper	94.9	88.0	73.3 - 103
iron	24400*	15700	6610 - 24900
lead	184	158	129 - 187
magnesium	3780*	2260	1760 - 2750
manganese	703	420	343 - 497
mercury	5.32	5.18	3.42 - 6.87
molybdenum	80.2	69.6	55.5 - 83.7
nickel	137	120	99.1 - 141
potassium	33000*	3000	2200 - 3800
selenium	146	130	101 - 159
silver	127	104	68.9 - 139
sodium	15600*	1080	692 - 1470
strontium	326	113	90.5 - 135
thallium	106	94.0	72.8 - 115
tin	175	149	104 - 194
titanium	3100*	284	116 - 453
vanadium	151	111	85.1 - 137
zinc	311	272	215 - 329

Method 3050 HNO3, H2O2 Parameter	Total Concentration [1] mg/Kg	Certified Value [2] mg/Kg	Performance Acceptance Limits™ [3] mg/Kg
aluminum	55600*	7380	4440 - 10300
antimony	160	75.2	D.L. - 198
arsenic	316	284	225 - 343
barium	869	217	177 - 257
beryllium	60.9	53.6	42.7 - 64.5
boron	129	89.5	58.9 - 120
cadmium	114	103	83.6 - 122
calcium	9750*	3540	2800 - 4270
chromium	249	224	172 - 275
cobalt	113	101	82.0 - 120
copper	94.9	85.5	70.4 - 100
iron	24400*	12500	5480 - 19500
lead	184	162	132 - 192
magnesium	3780*	2160	1650 - 2670
manganese	703	415	330 - 500
mercury	5.32	5.18	3.42 - 6.87
molybdenum	80.2	68.8	52.7 - 84.9
nickel	137	119	98.5 - 140
potassium	33000*	2840	2160 - 3520
selenium	146	135	104 - 166
silver	127	107	49.8 - 164
sodium	15600*	1010	709 - 1310
strontium	326	111	89.0 - 133
thallium	106	99.3	76.8 - 122
tin	175	148	70.6 - 225
titanium	3100*	283	104 - 463
vanadium	151	104	70.5 - 138
zinc	311	275	222 - 328

TITLE: TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010

Prepared By: George Brewer Date: 7/98

Approved By:

Group Supervisor: George Brewer Date: 01/23/01

Operations Manager: John C. Burton Date: 1/23/01

QA Officer: Deborah J. Nadeau Date: 1.23.01

General Manager: Duncan F. Hughes Date: 1/25/01

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
01 6010B	Format changes, added pollution prevention, expanded procedure and QC sections. Added tables.	DH	1.23.01	1/23/01
02 6010B	Calibration begins with analysis of S0 (cal. blank) followed by S1 (Mixed Cal. Std.) Changes to section 7.5 and Table 8 to reflect this. Made changes to element concs. in Tables 3, 4, 5, 6 to reflect current practices.	DH	10.21.02	10.21.02
03 6010B	Added MN-IEC to standards run. Changed frequency of LRS. Changed concentration % HNO_3 in calibration blank. CRI changed from three separate solutions to one. Changed CRI vendor.	MRC	04.15.04	04.15.04
04	Updated ICV, CCV, ICB, PQL Chk std. PBW, PBS, MS & MSD acceptance criteria updated Table 1	LAD	05/06	05/06
05	Updated Tables 3, 4, 5, 6 and 7 with current Standard concentrations and prep. Updated Table 1 with current practices including NAVY award findings. Updated Sections 2, 7.2, 7.6 and Table 1 with new ICP information. Updated Table 8 with current sequence requirements.	LAD	07/07	07/07

TITLE: TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
06	Added hardness definition and calculation (APP. 1)	LAD	09/07	09/07
07	Updated Summary to reflect new ICP functions. Removed ICP set-up updated tables to reflect changes in Standard Concentrations and preparation	LAD	11/08	11/08
08	Updates to Sections 8 and 10, Tables 1 and 2 to reflect changes from 6010B to 6010C. Added LLQC information and criteria to Sect. 8 and Table 1. Added criteria to analyze PQL standard at the beginning and END of each run.	LAD	02/09	02/09
09	Updated Sections 8, 9, 10 and Table 1 for compliance with DoD QSM version 4.1.	LAD	08/09	08/09
10	Added Table 2 - DoD QSM Ver. 4.1 QC Requirements. Minor correction to Table 1.	LAD	04/10	04/10
11	Added yttrium criteria to section 7 and Table 1.	LAD	06/10	06/10

TITLE: **TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010**

Please acknowledge receipt of this standard operating procedure by signing and dating both of the spaces provided. Return the bottom half of this sheet to the QA Department.

I acknowledge receipt of copy ___ of document **SOP CA-608-11**, titled **TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010.**

Recipient: _____ Date: _____

KATAHDIN ANALYTICAL SERVICES, INC.
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy ___ of document **SOP CA-608-11**, titled **TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010.**

Recipient: _____ Date: _____

TITLE: **TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010**

1.0 SCOPE AND APPLICATION

Inductively coupled plasma atomic-emission spectroscopy (ICP-AES) determines trace elements, including metals, in solution. The purpose of this SOP is to describe the procedures used by Katahdin Analytical Services, Inc. personnel to analyze aqueous and solid samples for trace metals by USEPA Method 6010 (Test Methods for Evaluating Solid Waste, Physical/ Chemical Methods, USEPA SW846).

Sample types that may be analyzed using these methods include drinking waters, ground waters, aqueous samples, TCLP, SPLP and EP Toxicity extracts, industrial and organic wastes, soils, sludges, sediments, and other solid wastes. The following elements may be analyzed under this SOP: Al, Sb, As, Ba, Be, B, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Mo, Ni, K, Se, Si, Ag, Na, Sn, Sr, Tl, Ti, V, and Zn.

All samples, except filtered ground water samples, analyzed under USEPA Method 6010 require digestion prior to analysis. USEPA Methods 3005, 3010, and 3050 describe appropriate digestion procedures for samples to be analyzed by ICP-AES under EPA Method 6010. Refer to current revisions of Katahdin SOPs CA-604 and CA-605, current revisions, for sample digestion procedures.

1.1 Definitions

Analytical Spike - An aliquot of a sample to which a known amount of analyte has been added before analysis and after digestion, if digestion is required.

CCB - Continuing Calibration Blank - An analyte-free solution consisting of acidified reagent water used to verify calibration accuracy periodically during analysis.

CCV - Continuing Calibration Verification - A midrange standard used to verify calibration accuracy periodically during analysis.

CRI - Contract Required detection limit sample for ICP - A low concentration standard used to verify calibration accuracy near the low end of the calibration range.

Duplicate - A second aliquot of a sample that is prepared and analyzed in the same way as the original sample in order to determine the precision of the method.

ICB - Initial Calibration Blank - An analyte-free solution consisting of acidified reagent water used to verify calibration accuracy.

ICP-AES - Inductively Coupled Plasma Atomic Emission Spectroscopy.

ICS - Interference Check Sample - Two standards (ICSA and ICSAB) used to verify the effectiveness of interelement correction and background correction. Solution ICSA contains only interferents (Al, Ca, Fe, and Mg) at high concentrations (200 to

500 mg/L); solution ICSAB contains interferents at the same concentrations as well as analytes at low (20 mg/L or less) concentrations.

ICV - Initial Calibration Verification - A standard made from a source independent from the calibration standards and with analyte concentrations different from those in the CCV; used to verify the accuracy of the instrument calibration.

IDL - Instrument Detection Limit - The lowest concentration of an analyte that can be determined with 99% confidence.

LOD – Limit of Detection – An estimate of the minimum amount of a substance that an analytical process can reliably detect. An LOD is analyte and matrix-specific and is used for DoD QSM acceptance criteria.

LOQ – Limit of Quantitation.- The minimum concentration of a target analyte that produces a quantitative result within specified limits of precision and bias.

LCS - Laboratory Control Sample - A standard or solid reference material that has been brought through the sample preparation process.

LRS - Linear Range Standard - A high-concentration standard used to determine the upper reporting limit of the ICP calibration.

PB - Preparation Blank - Reagent water that has been brought through the sample preparation process.

PQL - Practical Quantitation Limit - The lowest concentration of an analyte that is routinely reported by the laboratory; nominally three to five times the IDL.

Matrix Spike - An aliquot of a sample to which a known amount of analyte has been added before digestion.

Serial Dilution - The dilution of a sample by a factor of five. When corrected by the dilution factor, the measured analyte concentrations of the diluted sample should agree with those of the original undiluted sample within specified limits. Serial dilution may reflect the influence of interferents.

Hardness – The sum of the calcium and magnesium concentrations, both expressed as calcium carbonate, in mg/L.

1.2 Responsibilities

This method is restricted to use by, or under the supervision of, analysts experienced in ICP analysis by EPA Method 6010. Each analyst must demonstrate and document their ability to generate acceptable results with this method. Refer to Katahdin SOP QA-805, current revision, “Personnel Training & Documentation of Capability”.

It is the responsibility of all Katahdin technical personnel involved in ICP analysis by Method 6010 to read and understand this SOP, to adhere to the procedures outlined, and to properly document their data in the appropriate lab notebook. Any deviations from the test or irregularities with the samples should also be recorded in the lab notebook and reported to the Department Manager or designated qualified data reviewer responsible for this data.

It is the responsibility of the Department Manager to oversee that members of their group follow this SOP, to ensure that their work is properly documented and to initiate periodic review of the associated logbooks.

1.3 Safety

Users of this procedure must be cognizant of inherent laboratory hazards, proper disposal procedures for contaminated materials and appropriate segregation of hazardous wastes. The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical should be treated as a potential health hazard. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Everyone involved with the procedure must be familiar with the MSDSs for all the materials used in this procedure.

Each qualified analyst or technician must be familiar with Katahdin Analytical Environmental Health and Safety Manual including the Katahdin Hazardous Waste Plan and must follow appropriate procedures. These include the use of appropriate personal protective equipment (PPE) such as safety glasses, gloves and lab coats when working with chemicals or near an instrument and not taking food or drink into the laboratory. Each analyst should know the location of all safety equipment. Each analyst shall receive a safety orientation from their Department Manager, or designee, appropriate for the job functions they will perform.

Samples, sample digestates, standards, and other reagents used in ICP analysis may contain high concentrations of acids and toxic metals. Safety glasses should be worn when changing or adjusting argon tanks.

1.4 Pollution Prevention/Waste Disposal

Whenever possible, laboratory personnel should use pollution prevention techniques to address their waste generation. Refer to the current revision of the Katahdin Hazardous Waste Management Program for further details on pollution prevention techniques.

Wastes from ICP analysis should be disposed of in a manner appropriate to the hazards they present. Wastes generated during the preparation of samples must be disposed of in accordance with the Katahdin Analytical Environmental Health and Safety Manual I and SOP SD-903, “Sample Disposal,” current revision. Expired

standards are lab packed, placed in the Katahdin hazardous waste storage area, and disposed of in accordance with this SOP.

2.0 SUMMARY OF METHOD

This method describes multielemental determinations by ICP-AES using simultaneous optical systems and radial and axial viewing of the plasma. The basis of the method is the measurement of atomic emission from sample atoms entrained in an argon plasma by optical spectroscopy. Samples are nebulized and the aerosol that is produced is transported to the plasma torch where thermal excitation of entrained atoms and ions occurs. Characteristic atomic-line and ionic-line emission spectra are produced by a radio-frequency inductively coupled plasma (ICP). The spectra are dispersed by a grating and the intensities of the emitted lines are monitored by a solid state charge injection device (CID) camera system. Photocurrents from the CID camera system are measured by a computer system. Element concentrations of unknown samples are quantitated by comparison of sample emission intensities to emission intensities of standards of known concentration. A background correction technique is used to compensate for variable background contribution to the determination of trace elements. Background is measured adjacent to the analyte lines on samples during analysis. The position selected for the background intensity measurement, on either or both sides of the analytical line, has been determined by the complexity of the spectrum adjacent to the analytical line. The position used must be relatively free of spectral interference and must reflect the same change in background intensity as occurs at the analyte wavelength. Physical interferences are corrected through the use of an internal standard (yttrium) that is automatically added to all samples and standards prior to nebulization. The possibility of additional interferences (noted in section 3) must be recognized and appropriate corrections applied.

3.0 INTERFERENCES

Several types of interference effects may contribute to inaccuracies in the determination of trace elements. They can be summarized as spectral interferences, physical interferences, and chemical interferences.

Spectral interferences can be categorized as 1) overlap of a spectral line from another element; 2) unresolved overlap of molecular band spectra; 3) background contribution from continuous or recombination phenomena; and 4) background from stray light from the line emission of high concentration elements. The first of these effects is compensated by utilizing the computer correction of raw data, requiring the monitoring and measurement of the interfering element (interelement correction). The second effect is controlled by choosing analytical wavelengths that are free from overlapping molecular emission spectra. The third and fourth effects are usually compensated by a background correction adjacent to the analyte line. Uncorrected spectral interferences may be detected through examination of serial dilution and matrix spike data.

Physical interferences are generally considered to be effects associated with sample nebulization and transport processes. Such properties as changes in viscosity and surface tension can cause significant inaccuracies, especially in samples that may contain high dissolved solids and/or acid concentrations. Matrix matching of standards and samples and the use of a peristaltic pump may lessen these interferences. If these types of interferences are operative, they must be reduced by dilution of the sample and/or utilization of standard addition techniques. Another problem that can occur from high dissolved solids is salt buildup at the tip of the nebulizer. This affects aerosol flow rate causing instrumental drift. Regular cleaning of nebulizer tips and dilution of samples with high dissolved solids contents are used to control this problem. Physical interferences are also corrected by this laboratory through the use of an internal standard. Uncorrected physical interferences may be detected through examination of serial dilution and matrix spike data. Instrument drift caused by the salting up of nebulizer tips may also be detected by looking for oriented drift in calibration verification standards analyzed regularly throughout the run.

Chemical interferences are characterized by molecular compound formation, ionization effects, and solute vaporization effects. Normally these effects are not pronounced with the ICP technique; however, if observed they can be minimized by careful selection of operating conditions (i.e., incident power, observation position, etc.), by matrix matching, and by standard addition procedures. These types of interferences can be highly dependent on matrix type and the specific analyte element. Uncorrected chemical interferences may be detected through examination of serial dilution data.

4.0 APPARATUS AND MATERIALS

4.1 Computer-controlled inductively-coupled plasma atomic emission spectrometer (plasma viewed radially or axially) equipped for internal standardization, and capable of performing automatic background correction and interelement correction. For more information refer to the current revision of Katahdin SOP CA-632, “Operation and Maintenance of the Thermo ICAP 6500 ICP Spectrophotometer”.

4.2 Computer-controlled autosampler.

4.3 Argon gas supply – high purity.

4.4 Volumetric glassware of suitable precision and accuracy.

4.5 Automatic pipets of suitable precision and accuracy. Calibrated Eppendorf Reference pipets and Finn digital pipets are appropriate.

Refer to the appropriate instrument-specific SOP for additional required equipment.

5.0 REAGENTS

5.1 Hydrochloric acid, concentrated (HCl) – spectroscopic grade.

5.2 Nitric acid, concentrated (HNO_3) – spectroscopic grade.

5.3 Reagent water, trace metals free.

5.4 Calibration blank – reagent water containing HCl (5% v/v) and HNO_3 (5% v/v). Calibration blank solution is prepared in large volumes (up to 20 liters) and stored in a carboy. Calibration blank solution is used in establishing the analytical curve, and in all initial and continuing calibration blank determinations. This solution is also used to flush the system between standards and samples. Intermediate and working standards are prepared by diluting stock standards and intermediate standards with calibration blank solution so that all standards and blanks are acid matrix-matched to sample digestates.

5.5 Single element and multielement stock standard solutions – purchased standards prepared from high purity salts or metals, and supplied by the vendors with certificates of purity and analysis. Refer to Tables 3 and 4 for a listing of stock standards required, and to Table 8 for element concentrations in stock standards.

5.6 Intermediate standard solutions – laboratory-prepared multielement standards that are used in the subsequent preparation of working standards. Refer to Table 5 for a listing of intermediate standards required and for preparation instructions. Refer to Table 7 for element concentrations in intermediate standards.

5.7 Working standard solutions – laboratory-prepared multielement standards that are used to calibrate the instrument and to perform all necessary QC checks. Refer to Table 4 for a listing of working standards and for preparation instructions. Refer to Table 6 for element concentrations in working standards.

5.8 5 mg/L yttrium internal standard solution – add 0.5 mL 10000 mg/L yttrium stock standard to a 1000 mL volumetric flask half filled with calibration blank solution. Bring to volume with calibration blank solution.

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

Samples to be analyzed for trace metals by ICP should be collected and preserved as described in the following table.

Matrix	Container[1]	Volume / Weight	Preservation / Treatment	Holding Time
Aqueous (total)	P, G	250 mL	HNO_3 to pH < 2	6 months
Aqueous (dissolved)	P, G	250 mL	Filter, HNO_3 to pH < 2	6 months
Solid	P, G	10 g	Cool, 4°C	6 months

[1] P = polyethylene or, G = glass

7.0 PROCEDURES

7.1 Begin by following the startup and calibration instructions provided in the current revision of Katahdin SOP CA-632, "Operation and Maintenance of the Thermo ICAP 6500 ICP Spectrophotometer"

7.2 Analysis must proceed in the sequence described in Table 9 to ensure that all necessary quality control samples are analyzed at the appropriate frequencies. A minimum of two replicate integrations is required for all standards and samples. Analysis always begins with the analysis of a calibration blank solution (S0) followed by analysis of a multielement calibration standard (S1 in Table 4) to calibrate the instrument. The system is flushed with calibration blank for two minutes between each sample and standard, and each sample and standard is aspirated for one minute prior to the beginning of emission measurements.

7.3 Analysis continues with analysis of the initial calibration verification standard (ICV) and the initial calibration blank (ICB) to verify the accuracy of the calibration. Refer to Section 8 and Table 1 for additional information.

7.4 A continuing calibration verification standard (CCV) and a continuing calibration blank (CCB) must be analyzed at the beginning of the run, after every ten samples, and at the end of the run to verify the continued accuracy of the calibration. Refer to Section 8 and Table 1 for additional information.

7.5 Interference check standard solutions (ICSA and ICSAB) must be analyzed at the beginning, end, and at periodic intervals (4-6 hours, 30-40 analytical samples) throughout the sample run to verify the accuracy of the IEC factors. Refer to Section 8 and Table 1 for additional information.

7.6 A practical quantitation limit standard (PQL) must be analyzed at the beginning of each run to determine the accuracy of the calibration at the reporting limit. Refer to Section 8 and Table 1 for additional information.

7.7 All sample analytical results for a particular element that are bracketed (preceded or followed) by failing results in a QC sample (ICV, ICB, CCV, CCB, ICSA, or ICSAB) for that element must not be reported. The sample must be reanalyzed for the element in question.

7.8 All samples that exceed the linear dynamic range must be diluted and reanalyzed. This includes samples with interfering elements that exceed the calibration ranges, because accurate quantitation of interfering elements is necessary for reliable interelement correction. For example, if a sample has been submitted to the laboratory for lead analysis, and the measured aluminum concentration of that

sample exceeds the calibration range for aluminum, it must be diluted sufficiently to bring aluminum within the linear dynamic range and the lead result must be reported from that dilution analysis.

7.9 If dilutions of digested samples are performed, the measured element concentrations must be multiplied by the dilution factor prior to reporting. This is accomplished automatically by entering the dilution factor in the autosampler table prior to initiation of analysis.

7.10 All analyses are performed using yttrium as an internal standard to compensate for enhancement or depression of the analytical signal due to matrix effects. Yttrium solution is pumped at a constant rate through one channel of the peristaltic pump. Samples and standards are pumped through a second channel of the pump. The tubing carrying the internal standard is connected to the tubing carrying samples and standards downstream from the pump, and mixing of the two streams is accomplished in a mixing coil downstream from the connection, prior to nebulization. For each sample or standard, the computer that controls the spectrometer divides the detected emission signal for each element by the detected yttrium emission signal prior to quantitation, thus normalizing all emission signals to that of yttrium. The yttrium recovery must be within ± 20% of the counts of the initial calibration blank. If the recovery is outside of this, the sample must be diluted and reanalyzed.

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

USEPA Method 6010 requires the laboratory to perform specific quality control checks to assess laboratory performance and data quality. Minimum frequencies, acceptance criteria, and corrective actions for these control checks are tabulated in Table 1 and are described below. Table 1 criteria are intended to be guidelines for analysts. The table does not cover all possible situations. If any of the QC requirements are outside the recovery ranges listed in Table 1, all associated samples must be evaluated against all the QC. In some cases data may be reported, but may be reanalyzed in other cases. Making new reagents and standards may be necessary if the standardization is suspect. The corrective actions listed in Table 1 may rely on analyst experience to make sound scientific judgments. These decisions are based on holding time considerations and client and project specific Data Quality Objectives. The supervisor, Operations Manager, and/or Quality Assurance Officer may be consulted to evaluate data. Some samples may not be able to be reanalyzed within hold time. In these cases "qualified" data with narration may be advisable after consultation with the client.

In some cases the standard QC requirements listed in this section and in Table 1 may not be sufficient to meet the Data Quality Objectives of the specific project. Much of the work performed at the lab is analyzed in accordance with specific QC requirements spelled out in a project specific Quality Assurance Project Plan (QAPP) or in a program specific Quality Systems Manual (QSM). The reporting limits, acceptance criteria and/or corrective actions may be different than those specified in this SOP. In these cases the appropriate information

will be communicated to the Department Manager and/or senior chemists before initiation of the analyses so that specific product codes can be produced for the project. In addition, the work order notes for each project will describe the specific QAPP or QSM to be followed.

INITIAL DEMONSTRATION OF PERFORMANCE

8.1 Instrument detection limits (IDL) are determined quarterly for each analyte analyzed on each instrument. This determination requires seven replicate analyses of a reagent water spiked at 3-5 times the anticipated detection limit for each analyte, performed on three non-consecutive days. The standard deviation of the 21 analyses is multiplied by three to obtain the IDL. For more information on performing IDL determinations, refer to the current revision of Katahdin SOP QA-806.

8.2 Method detection limits (MDL) are determined annually for each analyte analyzed on each instrument. This determination requires at least seven replicate digestions and analyses of a reagent water spiked at 3-5 times the anticipated MDL for each analyte. MDLs differ from IDLs in that the seven replicates are digested prior to analysis, and they may be analyzed on a single day. The standard deviation of the 7 (or more) replicate analyses is multiplied by the Student's t-value to obtain the MDL. For more information on performing MDL determinations, refer to the current revision of Katahdin SOP QA-806.

8.3 Limits of Detection (LOD) are used when evaluating data using DoD QSM. The LOD is established by spiking a quality system matrix at 2-3 times the detection limit for a single analyte standard and 1-4 times the detection limit for a multi-analyte standard. The LOD must be verified quarterly. For more information on performing LOD determinations, refer to the current revision of Katahdin SOP QA-806.

8.4 Limits of Quantitation (LOQ) are used when evaluating data using DoD QSM. The LOQ must be above the LOD.

8.5 A Lower Limit of Quantitation Check (LLQC) sample must be prepared and analyzed annually or on an as-needed basis to confirm the laboratory's Practical Quantitation Limits (PQLs). The LLQC sample is equivalent to the PQL standard (Section 8.10) but is carried through the entire sample preparation and analysis process. Element recoveries for the LLQC sample must fall within 70% to 130% of the expected concentrations to confirm the previously established PQLs.

8.6 The upper limit of the linear dynamic range (LDR) must be established for each wavelength utilized. It must be determined from a linear calibration prepared in the normal manner using the established analytical operating procedure for the instrument. The LDR should be determined by analyzing succeedingly higher standard concentrations of the analyte until the observed analyte concentration differs by no more than 10% from the stated concentration of the standard. Determined LDRs must be documented and kept on file. The LDR which may be

used for the analyses of samples should be judged by the analyst from the resulting data. Determined sample analyte concentrations that are greater than the determined upper LDR limit must be diluted and reanalyzed. The LDRs should be verified **every six months** or whenever, in the judgment of the analyst, a change in analytical performance caused by either a change in instrument hardware or operating conditions would dictate they be redetermined.

8.7 The alkali and alkaline earth metals may have non-linear response curves due to ionization and self-absorption effects. These curves may be used for quantitation of samples if the effective range is checked and if the second order curve fit has a correlation coefficient of 0.998 or better. Third order fits are not acceptable. Non-linear response curves must be revalidated and recalculated every six months.

ANALYTICAL RUN QC SAMPLES

8.8 An Initial Calibration Verification (ICV) solution is analyzed after the initial calibration to check calibration accuracy. The ICV solution is prepared by combining compatible elements from a standard source different than that of the calibration standard and at concentrations within the linear working range of the instrument. The results of the ICV must fall within 90% to 110% of the expected values. If the ICV fails, result for the failing elements may not be reported from the run unless the ICV recovery is greater than 110% and the sample result is less than the PQL.

No results may be accepted for failing elements if DoD QSM acceptance criteria are being used.

8.9 Continuing Calibration Verification (CCV) solutions are analyzed after the initial calibration, after every ten samples, and at the end of the analytical run. The CCV solution is prepared using the same standards used for calibration at concentrations near the mid-point of the calibration curve. Results of the CCVs must fall within 90% to 110% of the expected values. If a CCV fails, results for the failing elements may not be reported from the run unless the CCV recovery is greater than 110% and the sample result is less than the PQL (less than reporting limit for DoD QSM). Also, for failing elements, all samples analyzed after the last passing CCV must be reanalyzed.

8.10 Calibration blank solution is analyzed after each ICV and CCV. A calibration blank that is analyzed after the ICV is called an Initial Calibration Blank (ICB). A calibration blank that is analyzed after a CCV is called a Continuing Calibration Blank (CCB). The absolute values of results of ICBs and CCBs must be less than the Practical Quantitation Level (PQL) for each element. If an ICB or a CCB fails, results for the failing elements may not be reported from the run until the problem is corrected and a passing ICB or CCB has been analyzed, with the following exception. If the result for a CCB or ICB is greater than the PQL, sample results that are less than the PQL or greater than or equal to ten times the measured CCB

concentration may be reported. Also, for failing elements, all samples analyzed after the last passing CCB must be reanalyzed, with the exception noted above.

If DoD QSM acceptance criteria are being used, the absolute values of results of ICBs and CCBs must be less than the Limit of Detection (LOD). If an ICB or a CCB fails, results for the failing elements may not be reported from the run until the problem is corrected and a passing ICB or CCB has been analyzed.

8.11 Interference check solutions ICSA and ICSAB (refer to Section 1.1) are analyzed at the beginning of each run to verify interelement correction factors and background correction. ICSA contains interferent elements (Al, Ca, Fe, and Mg) only, at concentrations of 200 mg/L to 500 mg/L. Results for interfering elements in the ICSA must fall within 80% to 120% of the expected values. Results for unspiked elements in ICSA must fall within ± PQL if the PQL is greater than 0.01 mg/L, within ± 2xPQL if the PQL is less than or equal to 0.01 mg/L. If DoD QSM acceptance criteria are being used, the absolute value of unspiked elements must be less than the LOD. ICSAB contains interferent elements at concentrations of 200 mg/L to 500 mg/L, and analytes at concentrations of 20 mg/L or less. Results for all elements (interferents and analytes) in ICSAB must fall within 80% to 120% of the expected values. If the ICSA or ICSAB fails, results for the failing elements may not be reported from the run until the problem is corrected and a passing ICSA or ICSAB has been analyzed.

8.12 A Practical Quantitation Limit (PQL) Check Standard or low level continuing calibration verification (LLCCV) is analyzed at the beginning (after the ICV and ICB samples) and at the end of each run. Element concentrations in this solution are at the laboratories practical quantitation limit. Element recoveries for the PQL check Standard must fall between 70-130% of the expected values. If the PQL Check Standard fails, the results for the failing elements may not be reported from the run, unless the PQL Check Standard recovery is greater than 130% and the samples results are less than the PQL.

If DoD QSM acceptance criteria are being used, recoveries must fall between 80-120%. If the PQL Check Standard fails, the results for the failing elements may not be reported from the run.

PREPARATION BATCH QC SAMPLES

8.13 Each digestion batch of twenty or fewer samples will contain a preparation blank and a laboratory control sample. Each batch will also contain one or more of the following QC samples: laboratory control sample duplicate, sample duplicate, matrix spike sample or matrix spike sample duplicate.

8.14 A preparation blank (PBW or PBS), consisting of reagent water carried through the same process as associated samples, is prepared with each digestion batch of twenty or fewer samples. The results of preparation blanks must be less than the

Practical Quantitation Level (PQL) for each element. For DoD QSM acceptance criteria the results must be less than ½ the PQL except for common contaminants which must be less than the PQL. If a preparation blank fails, results for the failing elements may not be reported from the digestion batch, and all associated samples must be redigested, with the following exception. If the result for a preparation blank is greater than the PQL (greater than ½ PQL for DoD), associated sample results that are less than the PQL (less than ½ PQL for DoD) or greater than or equal to ten times the measured preparation blank concentration may be reported.

8.15 A laboratory control sample (LCS), consisting of spiked reagent water or a solid reference material carried through the same process as associated samples, is prepared with each digestion batch of twenty or fewer samples. Results for laboratory control samples must fall within 80% to 120% of the expected value, unless vendor-supplied limits (for solid reference materials) or laboratory-generated statistical limits are available. If a laboratory control sample fails, results for the failing elements may not be reported from the digestion batch, and all associated samples must be redigested with the following exception. If the LCS fails high, sample results less than the PQL may be reported.

If DoD QSM acceptance criteria are being used, recovery for solid matrix samples must fall between 80% to 120% except for Ag, which must fall between 75% and 120%. Results may not be reported without a valid LCS and will be qualified and explained if reanalysis cannot be performed.

SAMPLE MATRIX QC SAMPLES

8.16 Matrix spiked duplicate samples are prepared at a minimum frequency of one per digestion batch. The recovery for each element in a spiked sample or spiked duplicate sample must fall within 75% to 125% of the actual value if the result for the unspiked sample is less than four times the amount of spike added. If one or both spike recoveries fail, the associated sample result must be flagged on the report of analysis. If DoD QSM acceptance criteria are being used, recoveries must be the same as stated for laboratory control samples.

The relative percent difference between sample duplicate, matrix spiked duplicate or LCS duplicate, is calculated as follows:

$$RPD\ (\%) = \frac{|D_1 - D_2|}{(|D_1 + D_2|)/2} \times 100$$

where: D_1 = sample result
D_2= duplicate sample result

A control limit of 20% RPD is applied to duplicate analysis if the original sample result is greater than 50X the IDL. If the matrix spike duplicate analysis fails, the associated sample result must be flagged on the report of analysis.

8.15 A serial dilution is analyzed to check for chemical or physical interferences. If the analyte concentration of a sample is sufficiently high (minimally, 50 x IDL or 50 x LOQ if using DoD QSM acceptance criteria), the measured concentration of a serial dilution (1:5 dilution) of the sample should agree within 90% to 110% of the original determination. The percent difference between the original sample and the serial dilution should be calculated as follows:

$$\text{Difference (\%)} = \frac{|L-S|}{S} * 100\%$$

where: L = Serial dilution result (corrected for dilution)
S = Original sample result

If the serial dilution analysis fails, a matrix interference should be suspected. The associated sample result should be flagged on the report of analysis or the sample should be reanalyzed at dilution to eliminate the interference.

For DoD QSM samples a Post-digestion Spike (PDS) addition must be performed if the serial dilution is not within acceptance criteria.

8.16 Post-digestion Spike (PDS) additions must be performed for DoD QSM samples if the serial dilution is not within acceptance criteria or if the analyte concentrations in all samples are less than 50x the LOD. The spike addition should produce a concentration that is between 10 and 100x the LOQ. The recovery of the PDS must be within 75-125%. If the PDS fails, all samples must be run by method of standard additions or appropriately flagged.

9.0 METHOD PERFORMANCE

The method detection limit (MDL) and the limit of detection (LOD) are defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero. The MDLs and LODs are determined prior to sample analysis per type of instrument and filed with the Inorganic Department Manager and with the QAO. Refer to the current revision of Katahdin SOP QA-806, Method Detection Limit and Instrument Detection Limit Studies, for procedures on determining the MDL.

Refer to the current revision of Method 6010 for other method performance parameters and requirements.

10.0 APPLICABLE DOCUMENTS/REFERENCES

Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, USEPA SW846, 3rd Edition, Final Updates I, II, IIA, IIB, III, IIIA, IIIB and IV, February 2007, Method 6010C.

TITLE: **TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010**

Department of Defense Quality Systems Manual for Environmental Laboratories (DOD QSM), Version 4.1, 04/22/09
The National Environmental Laboratory Accreditation Conference (NELAC) Standards, June 2003.

Katahdin SOP CA-101, Equipment Maintenance and Troubleshooting, current revision.

Katahdin SOP QA-806, Method Detection Limit and Instrument Detection Limit Studies, current revision.

List of Tables & Figures

TITLE: **TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010**

TABLE 1

QC REQUIREMENTS

Method	QC Sample	Minimum Frequency	Acceptance Criteria	Corrective Action
USEPA 6010	Initial Calibration, minimum 1 point plus a calibration blank.	Daily prior to sample analysis.	Correlation coefficient ® ≥ 0.998	Recalibrate
	Initial Calibration Verification (ICV), prepared from a second source.	Before beginning a sample run.	Recovery within ± 10% of true value.	1) Do not use results for failing elements unless the ICV > 110% and the sample < the PQL. 2) Investigate and correct 3) DoD: No samples may be run until calibration is verified
	Initial Calibration Blank (ICB)	Immediately after the ICV.	Absolute value of ICB < PQL.	1) Do not use results if ≥ PQL and 10x< CCB level. 2) Investigate and correct problem.
	Continuing Calibration Verification (CCV)	At beginning of run, after every 10 samples, and at end of run.	Recovery within ± 10% of true value.	1) Do not use results for failing elements unless the CCV > 110% and the sample < the PQL. 2) Investigate and correct problem.
	Continuing Calibration Blank (CCB)	After every 10 samples and at end of the run.	Absolute value of CCB < PQL.	1) Do not use results if ≥ PQL and < 10x CCB level. 2) Investigate and correct problem.
	Practical Quantitation Level Check Standard (PQL) (LLCCV)	At beginning and end of run.	Recovery within ± 30% of true value.	1) Do not use results for failing elements unless the ICV > 110% and the sample < the PQL. 2) Investigate and correct problem.
	Interference Check Solution A (ICSA)	At beginning and end of run.	For Al, Ca, Fe, and Mg, recovery within ± 20% of true value. For analytes not spiked, ± PQL, or, if PQL ≤ 0.01 mg/L, ± 2x PQL.	1) Do not use results for failing elements. 2) Investigate and correct problem.
	Interference Check Solution AB (ICSAB)	At beginning and end of run.	Recovery of each analyte within ± 20% of true value.	1) Do not use results for failing elements. 2) Investigate and correct problem.
	Preparation Blank (PBW/PBS)	One per digestion batch of 20 or fewer samples.	Less than PQL.	1) Investigate source of contamination. 2) Redigest and reanalyze all associated samples if sample concentration ≥ PQL and <10x the blank concentration.
	Laboratory Control Sample (LCSW/LCSS)	One per digestion batch of 20 or fewer samples.	Recovery within ± 20% of true value, unless vendor-supplied or statistical limits have been established.	1) Investigate source of problem. 2) Redigest and reanalyze all associated samples. 3) DoD: Flag specific analytes if samples cannot be reanalyzed.
	Matrix Spike Sample (S)	One per digestion batch of 20 or fewer samples.	Recovery ± 25% of true value, if sample < 4x spike added.	1) Flag results.

TITLE: **TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010**

TABLE 1 (cont)

QC REQUIREMENTS

Method	QC Sample	Minimum Frequency	Acceptance Criteria	Corrective Action
USEPA 6010 (cont.)	Matrix Spike Duplicate Sample (P) or sample duplicate	One per digestion batch of 20 or fewer samples.	Recovery ± 25% of true value, if sample < 4x spike added. RPD ≤20% for duplicate spikes and sample duplicates.	1) Flag results.
	Serial Dilution (L)	One per digestion batch.	If original sample result is at least 50x IDL, 5-fold dilution must agree within ± 10% of the original result. Flag result or dilute and reanalyzed sample to eliminate interference	Perform post digestion spike addition (PDS)
	Post-Digestion Spike Sample (A)	When dilution test fails or analyte concentration in all samples <50x LOD	Recovery within ± 25%.	Run associated samples by method of standard addition or flag results.
	Internal Standard	Every sample	± 20% (compared to the initial calibration blank)	Dilute sample and reanalyze.
	Instrument Detection Limit (IDL) Study	Quarterly.	IDL < MDL PQL > 2-3 * the IDL	1) Repeat IDL study. 2) Raise PQL.
	Method Detection Limit (MDL) Study	Refer to KAS SOP QA-806, "Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications", current revision.		
	Lower Limit of Quantitation Check (LLQC) Sample	Digest and analyze annually or as needed to confirm PQLs	70% - 130% of true value	Re-evaluate PQLs
	Linear Range Study	Every six months	Run succeedingly higher stds until recovery not within ± 10%. Use highest passing concentration as upper limit of linear range.	Only accept data to highest passing concentration until next linear range study.
	Limit of Detection (LOD) Determination	Quarterly	LOD = 1-4X MDL	Repeat LOD Determination
	Limit of Quantification (LOQ) Determination	Quarterly	LOQ > LOD	

TITLE: **TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010**

TABLE 2

DoD QSM VERSION 4.1 QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Demonstrate acceptable analytical capability	Prior to using any test method and at any time there is a significant change in instrument type, personnel, test method, or sample matrix.	QC acceptance criteria published by DoD, if available; otherwise, method-specified criteria.	Recalculate results; locate and fix problem, then rerun demonstration for those analytes that did not meet criteria.	NA.	This is a demonstration of analytical ability to generate acceptable precision and bias per the procedure in Appendix C. No analysis shall be allowed by analyst until successful demonstration of capability is complete.
LOD determination and verification	Refer to current revision of SOP QA-806				
LOQ establishment and verification	Refer to current revision of SOP QA-806				
Instrument detection limit (IDL) study (ICP only)	At initial set-up and after significant change in instrument type, personnel, test method, or sample matrix.	IDLs shall be ≤ LOD.	NA.	NA.	Samples may not be analyzed without a valid IDL.
Linear dynamic range or high-level check standard (ICP only)	Every 6 months.	Within ± 10% of true value.	NA.	NA.	
Initial calibration (ICAL) for all analytes ICP: minimum one high standard and a calibration blank	Daily ICAL prior to sample analysis.	If more than one calibration standard is used, $r \geq 0.995$.	Correct problem, then repeat ICAL.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be run until ICAL has passed.
Second source calibration verification (ICV)	Once after each ICAL, prior to beginning a sample run.	Value of second source for all analyte(s) within ± 10% of true value.	Correct problem and verify second source standard. Rerun ICV. If that fails, correct problem and repeat ICAL.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be run until calibration has been verified.

TITLE: **TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010**

TABLE 2 (cont)

DoD QSM VERSION 4.1 QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Continuing calibration verification (CCV)	ICP: within ± 10% of true value; GFAA: within ± 20% of true value; CVAA: within ± 20% of true value.	Correct problem, rerun calibration verification. If that fails, then repeat ICAL. Reanalyze all samples since the last successful calibration verification.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to all results for the specific analyte(s) in all samples since the last acceptable calibration verification.	After every 10 field samples and at the end of the analysis sequence.	Problem must be corrected. Results may not be reported without a valid CCV. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Low-level calibration check standard	Daily, after one-point ICAL.	Within ± 20% of true value.	Correct problem, then reanalyze.	Flagging criteria are not appropriate.	No samples may be analyzed without a valid low-level calibration check standard. Low-level calibration check standard should be less than or equal to the reporting limit.
Method blank	One per preparatory batch.	No analytes detected > ½ RL (> RL for common lab contaminants) and > 1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater). Blank result must not otherwise affect sample results. For negative blanks, absolute value < LOD.	Correct the problem. Report sample results that are <LOD or >10x the blank concentration. Reprepare and reanalyze the method blank and all associated samples with results > LOD and < 10x the contaminated blank result.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply B-flag to all results for the specific analyte(s) in all samples in the associated preparatory batch.	Problem must be corrected. Results may not be reported without a valid method blank. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Calibration blank	Before beginning a sample run, after every 10 samples, and at end of the analysis sequence.	No analytes detected > LOD. For negative blanks, absolute value < LOD.	Correct problem. Re-prep and reanalyze calibration blank. All samples following the last acceptable calibration blank must be reanalyzed.	Apply B-flag to all results for specific analyte(s) in all samples associated with the blank.	
Interference check solutions (ICS)	At the beginning of an analytical run.	ICS-A: Absolute value of concentration for all non-spiked analytes < LOD (unless they are a verified trace impurity from one of the spiked analytes); ICS-AB: Within ± 20% of true value.	Terminate analysis; locate and correct problem; reanalyze ICS, reanalyze all samples.	If corrective action fails, apply Q-flag to all results for specific analyte(s) in all samples associated with the ICS.	

TITLE: **TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010**

TABLE 2

DoD QSM VERSION 4.1 QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
LCS containing all analytes to be reported	One per preparatory batch.	Water and Soil: Recovery must be within + 20% of the true value	Correct problem, then reprep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch.	Problem must be corrected. Results may not be reported without a valid LCS. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Matrix spike (MS)	One per preparatory batch per matrix	For matrix evaluation, recovery must be within + 20% of the true value.	Examine the project-specific DQOs. If the matrix spike falls outside of DoD criteria, additional quality control tests are required to evaluate matrix effects.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met.	For matrix evaluation only. If MS results are outside the LCS limits, the data shall be evaluated to determine the source of difference and to determine if there is a matrix effect or analytical error.
Matrix spike duplicate (MSD) or sample duplicate	One per preparatory batch per matrix.	MSD: For matrix evaluation, recovery must be within + 20% of the true value. MSD or sample duplicate: RPD ≤ 20% (between MS and MSD or sample and sample duplicate).	Examine the project-specific DQOs. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met.	The data shall be evaluated to determine the source of difference.
Dilution test	One per preparatory batch.	If sample concentrations > 50 x LOQ, then the five-fold dilution must agree within ± 10% of the original measurement.	Perform post-digestion spike (PDS) addition.	Flagging criteria are not appropriate.	Only applicable for samples with concentrations > 50 x LOQ.
Post-digestion spike (PDS) addition	When dilution test fails or analyte concentration in all samples < 50 x LOD.	Recovery within 75-125%.	Run all associated samples in the preparatory batch by method of standard additions (MSA) or see flagging criteria.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met.	Spike addition should produce a concentration of 10 – 100 x LOQ.
Method of standard additions (MSA)	When matrix interference is confirmed.	NA.	NA.	NA.	Document use of MSA in the case narrative.
Results reported between DL and LOQ	NA.	NA.	NA.	Apply J-flag to all results between DL and LOQ.	

TITLE: **TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010**

TABLE 3

SUMMARY OF METHOD MODIFICATIONS

Topic	Katahdin SOP CA-608-11	Method 6010, current revision
Apparatus/Materials		
Reagents		
Sample preservation/ handling		
Procedures		
QC - Spikes		
QC - LCS		
QC - Accuracy/Precision		
QC - MDL		
QC - Calibration Blanks	Acceptance criteria employed for 6010: ± PQL	Acceptance criteria stated in 6010: less than 10% of PQL

TITLE: **TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010**

TABLE 4

PREPARATION OF CALIBRATION AND QUALITY CONTROL STANDARDS

Sample or Solution Name	Component Solution Name	Source of Component	Amount of Component Added per 100 mL Final Volume (mL)
Calibration Standard (STD1 or S1)	ICP- intermediate Standard	Lab Prepared (see Table 5)	10.0
	QCS 26	High Purity Standards	1.0
Initial Calibration Verification (ICV)	QCP-CICV-3	Inorganic Ventures	0.96
	1000 mg/L Si	Inorganic Ventures	0.98
	1000 mg/L Al	High Purity Standards	0.96
	IV-28	Inorganic Ventures	0.4
	1000 mg/L Sn	Inorganic Ventures	0.04
Interference Check Sample A (ICSA)	CLPP-ICS-A	Inorganic Ventures	10.0
Interference Check Sample AB (ICSAB)	CLPP-ICS-A	Inorganic Ventures	10.0
	CLPP-ICS-B4	Inorganic Ventures	1.0
	ICSAB-INT	Lab Prepared (see Table 5)	5.0
Continuing Calibration Verification (CCV)	ICP intermediate standard	Lab Prepared (see Table 5)	5.0
	QCS 26	High Purity Standards	0.5
Practical Quantitation Limit Sample (PQL)	PQL-INT	Lab Prepared (see Table 5)	1.0

TITLE: **TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010**

TABLE 5

PREPARATION OF INTERMEDIATE STANDARDS

Sample or Solution Name	Component Solution Name	Source of Component	Amount of Component Added per 100 mL Final Volume (mL)
PQL-INT	1000 mg/L B,Li,Sn,Sr, W, U	H-P or IV	1.0 each
	10000 mg/L K, Na	H-P or IV	1.0 each
	1000 mg/L Ni	High Purity Standards	0.4
	1000 mg/L Co	High Purity Standards	0.3
	1000 mg/L Cu,V,Zn	High Purity Standards	0.25 each
	1000 mg/L Si	High Purity Standards	2.0
	1000 mg/L Cr,Ti,Tl,Ag	High Purity Standards	0.15 each
	1000 mg/L Cd,Se, Mo	High Purity Standards	0.1 each
	10000 mg/L Al	High Purity Standards	0.3
	1000 mg/L As,Sb	High Purity Standards	0.08 each
	1000 mg/L Ba,Be,Mn,Pb	High Purity Standards	0.05 each
	10000 mg/L Ca,Mg	High Purity Standards	0.05 each
	10000 mg/L Fe	High Purity Standards	0.1
ICSAB-INT	10000 mg/L K,Na	H-P or IV	4.0 each
	10000 mg/L B, Li, Mo,Sr,Sn,Ti, W, U	High Purity Standards	1.0 each
	1000 mg/L Si	High Purity Standards	4.0

TITLE: **TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010**

TABLE 6

ELEMENT CONCENTRATIONS IN WORKING STANDARDS

	CONCENTRATION IN SOLUTION, mg/L								
Element	**STD1**	**ICV**	**PQL**	**ICSA**	**ICSAB**	**CCV**	**AL_IEC**	**FE_IEC**	**MN_IEC**
Aluminum	25	10	0.3	500	500	12.5	500		
Antimony	1	0.4	0.008		0.6	0.5			
Arsenic	1	0.4	0.008		0.1	0.5			
Barium	1	0.4	0.005		0.5	0.5			
Beryllium	1	0.4	0.005		0.5	0.5			
Boron	1	0.4	0.1		0.5	0.5			
Cadmium	1	0.4	0.01		1.0	0.5			
Calcium	25	10	0.05	500	500	12.5			
Chromium	1	0.4	0.015		0.5	0.5			
Cobalt	1	0.4	0.03		0.5	0.5			
Copper	1	0.4	0.025		0.5	0.5			
Iron	25	10	0.1	200	200	12.5		200	
Lead	1	0.4	0.005		0.05	0.5			
Lithium	1	0.4	0.1		0.5	0.5			
Magnesium	25	10	0.05	500	500	12.5			
Manganese	1	0.4	0.005		0.5	0.5			10
Molybdenum	1	0.4	0.01		0.5	0.5			
Nickel	1	0.4	0.04		0.5	0.5			
Potassium	25	13.6	1		20	12.5			
Selenium	1	0.4	0.01		0.05	0.5			
Silicon	1	0.4	0.2		2	0.5			
Silver	1	0.4	0.015		0.2	0.5			
Sodium	25	10	1		20	12.5			
Strontium	1	0.4	0.1		0.5	0.5			
Thallium	1	0.4	0.015		0.1	0.5			
Tin	1	0.4	0.1		0.5	0.5			
Titanium	1	0.4	0.015		0.5	0.5			
Tungsten	1	0.4	0.1		0.5	0.5			
Uranium	1	0.4	0.1		0.5	0.5			
Vanadium	1	0.4	0.025		0.5	0.5			
Zinc	1	0.4	0.025		1.0	0.5			

TITLE: **TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010**

TABLE 7

ELEMENT CONCENTRATIONS IN INTERMEDIATE STANDARDS

Element	CONCENTRATION IN SOLUTION, mg/L			
	ICP Intermed STD		PQL-INT	ICSAB-INT
Aluminum	240		30	
Antimony			0.8	
Arsenic			0.8	
Barium			0.5	
Beryllium			0.5	
Boron			10	10
Cadmium			1.0	
Calcium	240		5.0	
Chromium			1.5	
Cobalt			3.0	
Copper			2.5	
Iron	240		10	
Lead			0.5	
Lithium	10		10	10
Magnesium	240		5.0	
Manganese			0.5	
Molybdenum			1.0	10
Nickel			4.0	
Potassium	150		100	400
Selenium			1.0	
Silicon	250		20	40
Silver			1.5	
Sodium	240		100	400
Strontium	10		10	10
Thallium			1.5	
Tin	10		10	10
Titanium			1.5	10
Tungsten	10		10	10
Uranium	10		10	10
Vanadium			2.5	
Zinc			2.5	

TITLE: **TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010**

TABLE 8

ELEMENT CONCENTRATIONS IN STOCK STANDARDS

Element	CONCENTRATION IN SOLUTION, mg/L					
	IV-28	QCS-26	2007 ICS-1	CLPP-ICS-A	CLPP-ICS-B4	QCP-CICV-3
Aluminum	100	100		5000		
Antimony	100	100			60	
Arsenic	100	100			10	500
Barium	100	100			50	
Beryllium	100	100			50	
Boron	100	100	500			
Cadmium	100	100			100	250
Calcium	100	100		5000		
Chromium	100	100			50	
Cobalt	100	100			50	
Copper	100	100			50	
Iron	100	100		2000		
Lead	100	100			5	500
Lithium	100					
Magnesium	100	100		5000		
Manganese	100	100			50	
Molybdenum	100	100	300			
Nickel	100	100			100	
Potassium	1000	1000				
Selenium	100	100			5	500
Silicon	50	50	230			
Silver	100	100			20	
Sodium	100	100				
Strontium	100					
Thallium	100	100			10	500
Tin						
Titanium	100	100	1000			
Vanadium	100	100			50	
Zinc	100	100			100	

TITLE: **TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010**

TABLE 9

REQUIRED ANALYTICAL SEQUENCE

Sequence Number	Standard/Sample	Purpose
1	Blank (Calibration Blank)	Initial calibration
2	S1 (Calibration Standard)	Initial calibration
3	ICV (Initial Calibration Verification)	Check calibration accuracy
4	ICB (Initial Calibration Blank)	Check calibration accuracy
5	PQL (Practical Quantitation Level Sample)	Check calibration accuracy near PQL, repeat before final CCV, CCB
6	ICSA (Interference Check Solution A)	Verify accuracy of IEC factors, repeat before final CCV, CCB
7	ICSAB (Interference Check Solution AB)	Verify accuracy of IEC factors, repeat before final CCV, CCB
8	CCV (Continuing Calibration Verification)	Check calibration stability
9	CCB (Continuing Calibration Blank)	Check calibration stability
10-19	Analyze up to 10 samples	
20	CCV (Continuing Calibration Verification)	Check calibration stability
25	CCB (Continuing Calibration Blank)	Check calibration stability
…	Continue analyzing sequences of up to 10 samples, followed by a CCV and a CCB	

TITLE: **TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010**

ATTACHMENT 1

HARDNESS BY CALCULATION

As referenced in "Standard Methods for the Examination if Water and Wastewater," Methods 2340 A & B, Hardness Introduction and Hardness by Calculation, American Public Health Association, 18th Edition, Revised 1992, total hardness is the sum of the calcium and magnesium concentrations, both expressed as calcium carbonate, in milligrams per liter.

Once the calcium and magnesium concentrations have been determined by EPA methods 6010, 6020, 200.7 or 200.8, the total hardness of an aqueous sample may be calculated as follows:

$$\text{Total Hardness, mg equivalent } CaCO_3/L = 2.497\ (Ca, mg/L) + 4.118\ (Mg, mg/L)$$

The calcium hardness of an aqueous sample may also be calculated as follows:

$$\text{Calcium Hardness, mg equivalent } CaCO_3/L = 2.497\ (Ca, mg/L)$$

TITLE: DIGESTION AND ANALYSIS OF SOLID SAMPLES FOR MERCURY BY USEPA METHOD 7471

Prepared By: George Brewer Date: 12/97

Approved By:

Group Supervisor: George Brewer Date: 01/29/01

Operations Manager: Jak C. Burton Date: 1/29/01

QA Officer: Deborah J. Nadeau Date: 1.29.01

General Manager: Dennis F. Murphy Date: 1/29/01

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
02 7471A	Format changes, added pollution prevention, other minor changes to sections 7, 8 and QA Table.	DN	1.29.01	1/29/01
03 7471A	Changed LeeMAN PS200 Automated Mercury Analyzer to Cetac M6100 Mercury analyzer. Revised sect. 10 to show correct reference material. Removed fig. 2 Revised sect. 4.8, 5.7 and 8.9 to reflect current practises. minor changes through out	LAD	021605	021605
04 7471A	Sect. 5.9 and 5.10 - changed preparation of intermediate mercury standards from daily to monthly. Sect. 7.8 - removed calibration blanks (LCB/CCB). They are prepared in sect. 7.6. Added weighing of boiling chips for the prep blanks. Sect. 8.3 - Removed intermediate standards	LAD	03/08	03/08
05	Revised Sections 8 and 10, and Tables 1 and 2 to update compliance from method 7471A to method 7471B.	LAD	02/09	02/09
06	Added LOD definition. Updated sections 8, 9, 10 and Table 1 for DOD QSM version 4.1 compliance.	DN	08/09	08/09

TITLE: DIGESTION AND ANALYSIS OF SOLID SAMPLES FOR MERCURY BY USEPA METHOD 7471

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
07	Added Table 2 with DoD QSM Version 4.1 QC Requirements	LAD	04/10	04/10

TITLE: DIGESTION AND ANALYSIS OF SOLID SAMPLES FOR MERCURY BY USEPA METHOD 7471

Please acknowledge receipt of this standard operating procedure by signing and dating both of the spaces provided. Return the bottom half of this sheet to the QA Department.

I acknowledge receipt of copy ___ of document SOP CA-611-07, Titled Digestion and Analysis of Solid Samples for Mercury by USEPA Method 7471.

Recipient: _____ Date: _____

KATAHDIN ANALYTICAL SERVICES, INC.
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy ___ of document SOP CA-611-07, Titled Digestion and Analysis of Solid Samples for Mercury by USEPA Method 7471.

.

Recipient: _____ Date: _____

TITLE: DIGESTION AND ANALYSIS OF SOLID SAMPLES FOR MERCURY BY USEPA METHOD 7471

1.0 SCOPE AND APPLICATION

The purpose of this SOP is to describe the procedure used by Katahdin Analytical Services, Inc. personnel for the digestion and analysis solid samples for mercury using cold vapor atomic absorption spectrophotometry.

This method is applicable to the determination of mercury in soils, sediments, bottom deposits, and sludges under USEPA Method 7471 (Test Method for Evaluating Solid Wastes, USEPA SW 846, Third Edition).

1.1 Definitions

CCB - Continuing Calibration Blank - An analyte-free solution consisting of acidified laboratory reagent grade water used to verify calibration accuracy periodically during analysis.

CCV - Continuing Calibration Verification - A midrange standard used to verify calibration accuracy periodically during analysis.

Duplicate - A second aliquot of a sample that is prepared and analyzed in the same way as the original sample in order to determine the precision of the method.

ICB - Initial Calibration Blank - An analyte-free solution consisting of acidified laboratory reagent grade water used to verify calibration accuracy.

ICV - Initial Calibration Verification - A standard made from a source independent from the calibration standards and with analyte concentrations different from those in the CCV; used to verify the accuracy of the instrument calibration.

IDL - Instrument Detection Limit - The lowest concentration of an analyte that can be determined with 95% confidence by the instrument.

LCS - Laboratory Control Sample - A standard or solid reference material that has been brought through the sample preparation process.

LOD – Limit of Detection – An estimate of the minimum amount of a substance that an analytical process can reliably detect. An LOD is analyte and matrix-specific and is used for DoD QSM acceptance criteria.

PB - Preparation Blank - Laboratory reagent grade water that has been brought through the sample preparation process.

PQL - Practical Quantitation Limit - The lowest concentration of an analyte that is routinely reported by the laboratory; nominally three to five times the IDL.

Matrix Spike - An aliquot of a sample to which a known amount of analyte has been added before digestion.

MDL - Method Detection Limit - The minimum concentration of an analyte that can be identified, measured, and reported with 99% confidence that the analyte concentration is greater than zero.

1.2 Responsibilities

This method is restricted to use by, or under the supervision of analysts experienced in the analysis of mercury by USEPA Method 7471. Each analyst must demonstrate and document their ability to generate acceptable results with this method. Refer to Katahdin SOP QA-805, current revision, "Personnel Training & Documentation of Capability".

It is the responsibility of all Katahdin technical personnel involved in analysis of mercury by USEPA Method 7471 to read and understand this SOP, to adhere to the procedures outlined, and to properly document their data in the appropriate lab notebook. Any deviations from the test or irregularities with the samples should also be recorded in the lab notebook and reported to the Department Manager or designated qualified data reviewer responsible for this data.

It is the responsibility of the Department Manager to ensure that members of their group follow this SOP, to ensure that their work is properly documented, and to initiate periodic review of the associated logbooks.

1.3 Safety

Many of the samples and reagents used in cold vapor atomic absorption are toxic or corrosive. Gloves, safety glasses, lab coats, and other protective clothing should be worn whenever these materials are handled. Because of the toxic nature of mercury vapor, care must be taken to avoid its inhalation. The instrument exhaust fan must be in operation whenever the mercury analyzer is in use (the fan should never be shut off).

Users of this procedure must be cognizant of inherent laboratory hazards, proper disposal procedures for contaminated materials and appropriate segregation of hazardous wastes. The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical should be treated as a potential health hazard. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Everyone involved with the procedure must be familiar with the MSDSs for all the materials used in this procedure.

Each qualified analyst or technician must be familiar with Katahdin Analytical Environmental Health and Safety Manual including the Katahdin Hazardous Waste Plan and must follow appropriate procedures. These include the use of appropriate personal protective equipment (PPE) such as safety glasses, gloves and lab coats when working with chemicals or near an instrument and not taking food or drink into the laboratory. Each analyst should know the location of all safety equipment. Each analyst shall receive a safety orientation from their Department Manager, or designee, appropriate for the job functions they will perform.

1.4 Pollution Prevention/Waste Disposal

Whenever possible, laboratory personnel should use pollution prevention techniques to address there waste generation. Refer to the current revision of the Katahdin Hazardous Waste Management Plan for further details on pollution prevention techniques.

Samples, sample digestates, standards, and other reagents used in cold vapor atomic absorption may contain high concentrations of acids, mercury, and other toxic metals. They should be disposed of in a manner appropriate to the types of hazards they present. All digested mercury samples and standards and excess reagents and standards should be disposed of in the satellite waste container for corrosive wastes (labeled “Waste Stream A”) that is located in the Metals Prep lab. Further information regarding waste classification and disposal may be obtained by consulting the laboratory's Katahdin Analytical Environmental Health and Safety Manual and the Department Manager.

2.0 SUMMARY OF METHOD

The cold vapor atomic absorption technique is based on the absorption of radiation at 253.7 nm by mercury vapor. It relies on the volatility of elemental mercury at room temperature. During preparation, organic mercurials are oxidized and elemental mercury is ionized to Hg^{3+}. During instrumental analysis, mercuric ions are reduced to elemental mercury by the addition of stannous chloride. Elemental mercury is then aerated from solution and passes through a cell positioned in the path of a mercury spectrophotometer, where absorbance (peak height) is measured as a function of mercury concentration and recorded by the associated computer. The mercury vapor is then swept out of the instrument into an exhaust hood, where it is evacuated from the laboratory.

3.0 INTERFERENCES

In addition to inorganic forms of mercury, organic mercurials may be present in environmental samples. These organo-mercury compounds will not respond to the cold

vapor atomic absorption technique unless they are first broken down and converted to mercuric ions. The presence of undigested organo-mercurials in samples will result in a low bias for analytical results. Certain volatile organic materials will also non-specifically absorb radiation at the 253.7 nm analytical wavelength. The presence of such compounds may result in a high bias for analytical results. For these reasons, complete digestion using potassium permanganate is required for all environmental samples. Complete digestion is indicated by the persistence of the purple permanganate color (indicating the presence of excess permanganate) following digestion.

Samples that are high in chlorides may require additional permanganate to maintain a persistent purple color following digestion. During the oxidation step, chlorides are converted to free chlorine, which will absorb radiation at the 253.7 nm analytical wavelength. Any free chlorine thus generated will be present in the headspace of the digestion vessel following digestion. Because samples are poured into autosampler tubes prior to analysis by the mercury analyzer, any free chlorine present in the headspace of the digestion vessels is not sampled by the instrument and the analysis is free of chlorine interference.

4.0 APPARATUS AND MATERIALS

4.1 250 mL Pyrex media bottles with plastic screw caps, for use as digestion vessels.

4.2 Water bath capable of maintaining a constant temperature of 95^{o} C.

4.3 Analytical balance capable of weighing to 0.01 g.

4.4 Adjustable volume automatic pipettes - 2 to 20 uL, 10 to 100 uL, 100 to 1000 uL. Calibrated Eppendorf Reference pipets and Finn digital pipets are appropriate.

4.5 Repipetters (adjustable repeating pipetters with reservoirs) for dispensing concentrated nitric acid, concentrated sulfuric acid, and other reagents.

4.6 Spirit-filled thermometer, NIST-traceable, covering the range from 20^{o} to 110^{o} C, for monitoring the temperature of the water bath. Mercury-filled thermometers are not acceptable for use in the metals laboratory, due to the possibility of breakage and consequent contamination.

4.7 Disposable graduated polystyrene sample cups, 200 mL capacity.

4.8 CETAC M6100 Mercury Analyzer and associated peripherals and parts.

Refer to Katahdin SOP CA-629, current revision, “Operation and Maintenance of the CETAC M6100 Mercury Analyzer” for additional required materials.

5.0 REAGENTS

5.1 Laboratory reagent grade water – mercury-free water.

5.2 Concentrated nitric acid (HNO_3), trace metal grade

5.3 Concentrated hydrochloric acid (HCl), trace metal grade

5.4 Aqua regia: Prepare an appropriate amount immediately before use by carefully adding three volumes of concentrated HCl to one volume of concentrated HNO_3 in a heat-proof beaker or flask. Preparation of aqua regia must be performed in a fume hood.

5.5 Potassium permanganate solution, 5% w/v: Dissolve 50 g of potassium permanganate in 1 L laboratory reagent grade water. The source reagent should be labeled as suitable for use in mercury determination.

5.6 Sodium chloride – hydroxylamine hydrochloride solution: Dissolve 120 g sodium chloride and 120 g hydroxylamine hydrochloride in laboratory reagent grade water and dilute to a final volume of 1 L.

5.7 Stannous chloride solution: Add 70 mL concentrated hydrochloric acid to 500 mL of laboratory reagent grade water. Add 100 g stannous chloride and bring to a final volume of 1 L. Mix to dissolve. Reagent should be labeled as suitable for use in mercury determination.

5.8 Mercury Stock Standards: Two 10.0 mg/L mercury stock standards, obtained from separate sources, are required. The mercury concentrations of these standards must be certified by the manufacturers as traceable to NIST reference standards.

5.9 Intermediate Mercury Standard A: Appropriately dilute a mercury stock standard to obtain a solution containing 1000 ug of mercury per liter in 2% nitric acid. This intermediate standard is used to prepare calibration standards, matrix spikes, CCVs, and laboratory control samples (refer to Section 8). The identity of the stock standard currently used to prepare this intermediate standard and instructions for its dilution may be obtained by consulting the Standards Preparation Logbook maintained in the Section. Intermediate Mercury Standard A must be prepared monthly, and disposed of appropriately after use. (Note: the concentrations of all stock standards must be certified by the vendors as traceable to NIST reference materials).

5.10 Intermediate Mercury Standard B: Appropriately dilute a mercury stock standard to obtain a solution containing 1000 ug of mercury per liter in 2% nitric acid. The source of the stock standard used to prepare Intermediate Mercury Standard B must be distinct from that used to prepare Intermediate Mercury Standard A (i.e. obtained

from a separate vendor). Intermediate Mercury Standard B is used to prepare the ICV (refer to Section 8.0). The identity of the stock standard currently used to prepare this intermediate standard and instructions for its dilution may be obtained by consulting the Standards Preparation Logbook maintained in the Section. Intermediate Mercury Standard B must be prepared monthly, and disposed of appropriately after use.

5.11 Solid Reference Material: A soil with a known or empirically-established mercury concentration for use in preparing the laboratory control sample for soils. Solid reference materials should be purchased with certificates listing reference values and quality control acceptance limits. See Figure 3 for an example certificate of analysis for a solid reference material.

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

Soil samples to be analyzed for mercury should be collected and preserved as described in the following table.

Matrix	Container[1]	Collection Volume/ Weight	Preservation/ Treatment	Holding Time
Solid	P, G	40 g	Cool to 4°C ± 2°	28 days

[1] P = polyethylene, G = glass

7.0 PROCEDURES

BOTTLE PREPARATION

7.1 Mercury digestion bottles are reused, and must be cleaned between uses. After the previous contents of the bottles have been discarded, bottles are segregated according to whether the measured mercury concentrations of the previous contents were above the PQL (contaminated bottles) or below the PQL (uncontaminated bottles). Labels are removed from the bottles by wiping with a paper towel saturated with toluene. Both contaminated and uncontaminated bottles are then cleaned with Liquinox and water, if necessary, to remove visible grime, and rinsed thoroughly with tap water.

7.2 Uncontaminated bottles are then triple-rinsed with laboratory reagent grade water, and are ready for reuse.

7.3 Contaminated bottles are placed in a bath containing 10% HCl for at least 12 hours. After acid-leaching, these bottles are triple rinsed with laboratory reagent grade water, and are then ready for reuse.

PREPARATION OF STANDARDS, QC SAMPLES, AND BLANKS

7.4 Prior to performing the digestion, make a list of the samples that are to be digested. Enter digestion information (Katahdin Sample Numbers, Bottle IDs, QC Batch ID, preparation date, analyst initials, etc.) into the ACCESS computer database and print out a copy of the benchsheet. All necessary details of sample preparation (standards preparation information, digestion times, digestion temps, initial weights and final volumes, pertinent observations, etc.) must be recorded on this benchsheet, which will be bound in the Mercury Preparation Logbook. Refer to Figure 1 for an example page from the Mercury Preparation Logbook.

7.5 Using a silver paint marker, label clean digestion bottles with the appropriate sample numbers and standard identifications for each sample and standard to be digested.

7.6 Using calibrated adjustable pipettes, prepare calibration standards by adding 0 uL, 20 uL, 50 uL, 100 uL, 500 uL, and 1000 uL of Intermediate Mercury Standard A to separate appropriately-labeled digestion bottles. The mercury concentrations of these calibration standards will be, respectively, 0 ug/L (calibration blank), 0.2 ug/L, 0.5 ug/L, 1.0 ug/L, 5.0 ug/L, and 10.0 ug/L. The 0 ug/L, 0.2 ug/L and 0.5 ug/L standards are analyzed during analysis as the CCB, PQL standard and the CCV (refer to Section 8.0), respectively, as well as being used in the creation of the calibration curve.

7.7 Using a calibrated adjustable pipette, prepare the initial calibration verification (ICV) standard (refer to Section 8) by adding 600 uL of Intermediate Mercury Standard B to an appropriately labeled digestion bottle. The mercury concentration of the ICV will be 6.0 ug/L.

7.8 Prepare an appropriate number of preparation blanks (PBS) by adding 1.0 g of Teflon boiling chips to labeled digestion bottles.

7.9 Prepare an appropriate number of laboratory control samples (LCSS) by weighing appropriate masses of solid reference material into labeled digestion bottles. The mercury concentration of these LCSSs will depend on the solid reference material used, and the mass of each aliquot. Refer to Figure 3 for an example certificate of analysis for a solid reference material.

7.10 Matrix spikes are prepared by adding 100 uL of Intermediate Mercury Std A to each matrix spike sample. The amount of mercury added to each matrix spike increases the final digestate concentration by 1.0 ug/L.

7.11 All calibration standards, QC samples, and blanks are digested in the same manner as client samples. Refer to Sample Preparation and Digestion, Steps 7.12 through 7.16 of this SOP.

SAMPLE PREPARATION AND DIGESTION

7.12 Weigh three approximate 0.2 g portions (a total of approximately 0.6 g) of untreated sample from different parts of the sample container and place them in the bottom of a labeled digestion bottle. The purpose of using three portions is to obtain a representative sample from the sample container.

7.13 Add 5 mL of laboratory reagent grade water and 5 mL of aqua regia to each sample, standard, and QC sample. Place bottles in a water bath located in a fume hood and heat for 2 minutes at 95^{o} C. Remove the bottles from the water bath and allow them to cool in a fume hood.

7.14 Add 50 mL of laboratory reagent grade water and 15 mL of potassium permanganate solution to each digestion bottle, swirl to mix, and allow to stand for at least 15 minutes. Samples that contain large amounts of oxidizable organic matter may require additional 15 mL aliquots of potassium permanganate solution. This is indicated by the failure of the purple permanganate color to persist for the entire 15 minute waiting period. Add additional 15 mL aliquots to samples as necessary until the purple color persists for 15 minutes. If any of the samples requires these additional aliquots of permanganate, note that fact on the mercury preparation benchsheet and accordingly adjust the final volumes recorded on the benchsheet for those samples.

When a persistent purple color has been obtained for all samples, place the digestion bottles in the water bath and heat for 30 minutes at 95^{o} C. Record initial and final time and temperatures on the mercury preparation benchsheet.

7.15 Remove the bottles from water bath and allow them to cool in a fume hood. If any of the samples have become colorless during heating, add additional 15 mL aliquots of potassium permanganate solution as necessary to obtain a persistent purple color and heat for an additional 30 minutes at 95^{o} C. Record any information regarding additional permanganate aliquots on the mercury preparation benchsheet and accordingly adjust the final volumes recorded on the benchsheet for the samples affected.

7.16 Add 6 mL of sodium chloride – hydroxylamine hydrochloride solution to each digestion bottle and swirl to mix. Perform this addition in a fume hood, as chlorine gas may be evolved. This will reduce the excess permanganate, and the sample will change from purple to colorless. Add 50 mL of laboratory reagent grade water to each bottle. Wait at least 30 seconds before proceeding with analysis.

INSTRUMENTAL ANALYSIS

7.17 Digested mercury samples are analyzed using the CETAC M6100 Mercury Analyzer. Analysis is automated and is controlled by the QuickTrace software running on a dedicated PC. Detailed instructions for setting up the instrument and running samples are given Katahdin SOP CA-629, "Operation and Maintenance of the CETAC M6100 Mercury Analyzer". The following information specifically pertains to analysis of digested samples in accordance with USEPA Method 7471, and should be used in conjunction with the instructions given in Katahdin SOP CA-629.

7.18 Instrument operating conditions and quality control acceptance limits are specified in the instrument software in "templates". The template that is used to analyze digested samples in accordance with USEPA Method 7471 is named "SW846-7470-7471".

7.19 Prior to analysis, digested samples, standards, and QC samples are decanted into autosampler tubes which are placed in racks on the instrument's autosampler. The "standards" autosampler rack has 10 positions for 25 x 100 mm autosampler tubes (50 mL capacity). Tubes containing the calibration standards, the ICV, the CCV, the ICB/CCB, and the PQL standard are placed in the appropriately labeled positions in this autosampler rack.

7.20 Client samples, batch QC samples (preparation blanks and laboratory control samples), and matrix QC samples (duplicates and matrix spikes) are decanted into 17 x 100 mm autosampler tubes (15 mL capacity), which are placed in the one of the "samples" autosampler racks. The "samples" autosampler racks have 60 positions for 17 x 100 mm autosampler tubes. Instructions for filling the "samples" autosampler racks, including recording the rack position of each sample, are contained in Katahdin SOP CA-629, "Operation and Maintenance of the CETAC M6100 Mercury Analyzer".

METHOD OF STANDARD ADDITIONS

7.21 The standard addition technique involves adding known amounts of standard to one or more aliquots of the processed sample solution. This technique compensates for a sample constituent that enhances or depresses the analyte signal, thus producing a different slope from that of the calibration standards. It will not correct for additive interferences that cause a baseline shift. The method of standard additions shall be used for analysis of all EP extracts, on all analyses submitted as part of a delisting petition, and whenever a new sample matrix is being analyzed.

7.21.1 The simplest version of this technique is the single-addition method, in which two identical aliquots of the sample solution, each of volume V_x, are taken. To the first (labeled A) is added a known volume V_S of a standard analyte solution of concentration C_S. To the second aliquot (labeled B) is

added the same volume V_S of the solvent. The analytical signals of A and B are measured and corrected for nonanalyte signals. The unknown sample concentration C_x is calculated:

$$C_X = \frac{S_B V_S C_S}{(S_A - S_B) V_X}$$

where S_A and S_B are the analytical signals (corrected for the blank) of solutions A and B, respectively. V_s and C_s should be chosen so that S_A is roughly twice S_B on the average, avoiding excess dilution of the sample. If a separation or concentration step is used, the additions are best made first and carried through the entire procedure.

7.21.2 Improved results can be obtained by employing a series of standard additions. To equal volumes of the sample are added a series of standard solutions containing different known quantities of the analyte, and all solutions are diluted to the same final volume. For example, addition 1 should be prepared so that the resulting concentration is approximately 50 percent of the expected absorbance from the endogenous analyte in the sample. Additions 2 and 3 should be prepared so that the concentrations are approximately 100 and 150 percent of the expected endogenous sample absorbance. The absorbance of each solution is determined and then plotted on the vertical axis of a graph, with the concentrations of the known standards plotted on the horizontal axis. When the resulting line is extrapolated to zero absorbance, the point of interception of the abscissa is the endogenous concentration of the analyte in the sample. The abscissa on the left of the ordinate is scaled the same as on the right side, but in the opposite direction from the ordinate. An example of a plot so obtained is shown in Figure 2. A linear regression program may be used to obtain the intercept concentration.

7.21.3 For the results of this MSA technique to be valid, the following limitations must be taken into consideration:

- The apparent concentrations from the calibration curve must be linear over the concentration range of concern. For the best results, the slope of the MSA plot should be nearly the same as the slope of the standard curve. If the slope is significantly different (greater than 20%), caution should be exercised.

- The effect of the interference should not vary as the ratio of analyte concentration to sample matrix changes, and the standard addition should respond in a similar manner as the analyte.

- The determination must be free of spectral interference and corrected for nonspecific background interference.

DATA REDUCTION AND REPORTING

7.22 Results are obtained in units of ug/L in the digestate. Results that exceed the calibration range of the instrument may not be reported - the sample must be appropriately diluted and reanalyzed. Results for diluted samples must be multiplied by the dilution factor prior to reporting. If additional aliquots of potassium permanganate were added during digestion, the change in digestate final volume must be taken into account in calculating the final result. Mercury results for solid samples are reported in units of ug/g, calculated on a dry weight basis. Calculation of mercury results for solid samples is performed automatically by the Metals reporting database, as follows:

$$\text{Mercury Concentration in Solid (mg/kg dry wt.)} = \frac{(C) \times (DF) \times (FV) \times 100}{(W) \times (TS)}$$

where C = Measured digestate concentration (ug/L)
DF = Instrument dilution factor
FV = Digestate final volume (L)
W = Digested wet sample weight (g)
TS = Total Solids (%)

7.23 Results are reported down to the laboratory's practical quantitation level (PQL), unless otherwise requested. Results below the PQL should be reported as "<PQL".

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

USEPA Method 7471 requires the laboratory to perform specific quality control checks to assess laboratory performance and data quality. Minimum frequencies, acceptance criteria, and corrective actions for these control checks are tabulated in Table 1 and are described below. Preparation instructions and the resulting mercury concentrations for calibration standards, QC standards, and matrix spikes are detailed in Sections 7.6 through 7.10 of this SOP. Table 1 criteria are intended to be guidelines for analysts. The table does not cover all possible situations. If any of the QC requirements are outside the recovery ranges listed in Table 1, all associated samples must be evaluated against all the QC. In some cases data may be reported, but may be reanalyzed in other cases. Making new reagents and standards may be necessary if the standardization is suspect. The corrective actions listed in Table 1 may rely on analyst experience to make sound scientific judgments. These decisions are based on holding time considerations and client and project specific Data Quality Objectives. The supervisor, Operations Manager, General Manager and/or Quality Assurance Officer may be consulted to evaluate data. Some samples

may not be able to be reanalyzed within hold time. In these cases "qualified" data with narration may be advisable after consultation with the client.

Much of the work performed at the lab is analyzed in accordance with specific QC requirements spelled out in a project specific Quality Assurance Project Plan (QAPP) or in a program specific Quality Systems Manual (QSM). The reporting limits, acceptance criteria and/or corrective actions may be different than those specified in this SOP. In these cases the appropriate information will be communicated to the Department Manager and/or senior chemists before initiation of the analyses so that specific product codes can be produced for the project. In addition, the work order notes for each project will describe the specific QAPP or QSM to be followed.

INITIAL DEMONSTRATION OF PERFORMANCE

8.1 Instrument detection limits (IDL) are determined quarterly for each analyte analyzed on each instrument by each method. This determination requires seven replicate analyses of laboratory reagent grade water spiked, performed on three non-consecutive days. The standard deviation of the 21 analyses is multiplied by three to obtain the IDL. For more information on performing IDL determinations, refer to the current revision of Katahdin SOP QA-806.

8.2 Method detection limits (MDL) are determined annually for each analyte analyzed on each instrument. This determination requires at least seven replicate digestions and analyses of laboratory reagent grade water spiked at 3-5 times the anticipated MDL for each analyte. MDLs differ from IDLs in that the replicates are digested prior to analysis, and they may be analyzed on a single day. The standard deviation of the 7 (or more) replicate analyses is multiplied by the Student's t-value to obtain the MDL. For more information on performing MDL determinations, refer to the current revision of Katahdin SOP QA-806.

8.3 Limits of Detection (LOD) are used when evaluating data using DoD QSM. The LOD is established by spiking a quality system matrix at 2-3 times the detection limit for a single analyte standard and 1-4 times the detection limit for a multi-analyte standard. The LOD must be verified quarterly. For more information on performing LOD determinations, refer to the current revision of Katahdin SOP QA-806.

ANALYTICAL RUN QC

8.4 Instrument calibration - The instrument must be calibrated each time it is set up, and calibration standards must be digested each day that samples are digested. Calibration includes analysis of a calibration blank and five calibration standards with graduated concentrations in the appropriate range. The concentration of one of the calibration standards must be at the Practical Quantitation Level (PQL). The correlation coefficient for the calibration curve must be at least 0.995. If the

calibration curve does not pass this test, analysis must be halted, the problem corrected, and the instrument recalibrated.

8.5 An Initial Calibration Verification (ICV) solution is analyzed after the initial calibration to check calibration accuracy. The ICV solution is prepared from a standard source different than that of the calibration standard and at a concentration within the working range of the instrument. The result of the ICV must fall within 90% to 110% of the expected value. If the ICV fails, results may not be reported from the run until the problem is corrected and a passing ICV has been analyzed.

8.6 The Continuing Calibration Verification (CCV) solution is analyzed after the initial calibration, after every ten samples, and at the end of the analytical run. The CCV solution is prepared using the same standard used for calibration at a concentration near the mid-point of the calibration curve. Results of the CCVs must fall within 90% to 110% of the expected value. If a CCV fails, associated sample results may not be reported from the run until the problem is corrected and a passing CCV has been analyzed. Also, all samples analyzed after the last passing CCV must be reanalyzed. For DoD QSM acceptance criteria, samples that are below the reporting limit may be reported if the CCV reads greater than 120%.

8.7 A calibration blank is analyzed after each ICV and CCV. A calibration blank that is analyzed after the ICV is called an Initial Calibration Blank (ICB). A calibration blank that is analyzed after a CCV is called a Continuing Calibration Blank (CCB). The absolute values of results of ICBs and CCBs must be less than the Practical Quantitation Level (PQL) for each element. If samples are being run using DoD QSM criteria, the absolute values of ICBs and CCBs must be less than the Limit of Detection (LOD). If an ICB or a CCB fails, results for the failing elements may not be reported from the run until the problem is corrected and a passing ICB or CCB has been analyzed. Also, all samples analyzed after the last passing CCB must be reanalyzed.

8.8 A standard with a mercury concentration that is at the Practical Quantitation Limit (PQL) is analyzed at the beginning of the run to determine calibration accuracy at the reporting limit. Result of the PQL standard should fall within 70% to 130% of the expected values. No corrective action has been established at this time.

PREPARATION BATCH QC SAMPLES

8.9 Preparation blank (PBW or PBS), consisting of reagent water carried through the same process as associated samples, is prepared with each digestion batch of twenty or fewer samples. The results of preparation blanks must be less than the Practical Quantitation Level (PQL) for each element. For DoD QSM acceptance criteria the results must be less than ½ the PQL except for common contaminants which must be less than the PQL. If a preparation blank fails, results for the failing elements may not be reported from the digestion batch, and all associated samples

must be redigested, with the following exception. If the result for a preparation blank is greater than the PQL (greater than ½ PQL for DoD), associated sample results that are less than the PQL (less than ½ PQL for DoD) or greater than or equal to ten times the measured preparation blank concentration may be reported.

8.10 A laboratory control sample (LCSS), consisting of solid reference material carried through the same process as associated samples, is prepared with each digestion batch of twenty or fewer samples. If a laboratory control sample fails, results may not be reported from the digestion batch, and all associated samples must be redigested. The laboratory uses a reference value and statistical acceptance limits for laboratory control samples are supplied by the vendor of the solid reference material. If samples are being prepared using DoD QSM acceptance criteria, the results of the LCSS must be within 80% - 120%.

SAMPLE MATRIX QC SAMPLES

8.11 Matrix spiked duplicate samples are prepared at a minimum frequency of one per digestion batch. Matrix spike recoveries for these samples are calculated as follows:

$$\text{Recovery (\%)} = \frac{(P - S)}{A} \times 100\%$$

where:

P = Spiked sample value
S = Original sample value
A = Spike amount

The recovery for each element in a spiked sample or spiked duplicate sample must fall within 75% to 125% of the actual value if the result for the unspiked sample is less than four times the amount of spike added. If one or both spike recoveries fail, a matrix interference should be suspected and the associated sample result should be flagged on the report of analysis. If DoD QSM acceptance criteria are being used, recoveries must be the same as stated for laboratory control samples.

The relative percent difference between matrix spiked duplicate sample results is calculated as follows:

$$\text{RPD (\%)} = \frac{|D_1 - D_2|}{(|D_1 + D_2|)/2} \times 100$$

where: D_1 = Spike sample result
D_2= Spike duplicate sample result

A control limit of 20% RPD is applied to matrix spike duplicate analysis. If the matrix spike duplicate analysis fails, the associated sample result should be flagged on the report of analysis.

9.0 METHOD PERFORMANCE

The method detection limit (MDL) and the limit of detection (LOD) are defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero. The MDLs and LODs are determined prior to sample analysis per type of instrument and filed with the Inorganic Department Manager and with the QAO. Refer to the current revision of Katahdin SOP QA-806, Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications, for procedures on determining the MDL.

Refer to the current revision of Method 7471 for other method performance parameters and requirements.

10.0 APPLICABLE DOCUMENTS/REFERENCES

Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, USEPA SW846, 3rd Edition, Final Updates I, II, IIA, IIB, III, IIIA, IIIB and IV, February 2007, Method 7471B.

Department of Defense Quality Systems Manual for Environmental Laboratories (DOD QSM), Version 4.1, 04/22/09.

The National Environmental Laboratory Accreditation Conference (NELAC) Standards, June 2003.

Katahdin SOP CA-101, Equipment Maintenance and Troubleshooting, current revision.

Katahdin SOP QA-806, Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications.

QuickTrace M6100 Mercury Analyzer Operator Manual Version 1.0.1, CETAC Technologies

QuickTrace Mercury Analyzer Software Manual, CETAC Technologies.

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TABLE 1

QC REQUIREMENTS

Parameter/ Method	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Mercury/ USEPA Method 7471B	Initial Calibration, 5 points plus a calibration blank.	Daily prior to sample analysis.	Correlation coefficient ≥ 0.995.	Correct problem and repeat calibration.
	Initial Calibration Verification (ICV), prepared from a second source.	Before beginning a sample run.	Recovery within ± 10% of true value.	Correct problem and repeat calibration.
	Initial Calibration Blank (ICB)	Before beginning a sample run.	Less than PQL.	Correct problem and repeat calibration.
	Practical Quantitation Level Standard (PQL)	Before beginning a sample run.	Recovery within ± 30% of true value.	No corrective action required at this time.
	Continuing Calibration Verification (CCV)	At beginning or run, after every 10 samples, and at end of the run	Recovery within ± 10% of true value	Repeat calibration and reanalyze all samples analyzed since the last successful CCV.
	Continuing Calibration Blank (CCB)	At beginning or run, after every 10 samples, and at end of the run	Less than PQL.	Repeat calibration and reanalyze all samples analyzed since the last successful CCB.
	Preparation Blank (PBS)	One per digestion batch of 20 or fewer samples.	Less than PQL.	1) Investigate source of contamination. 2) Redigest and reanalyze all associated samples if sample concentration ≥ PQL and < 10x the blank concentration.
	Laboratory Control Sample (LCSS)	One per digestion batch of 20 or fewer samples.	Recovery within vendor-supplied acceptance limits.	Redigest all affected samples.
	Matrix Spike Sample (S)	One per digestion batch of 20 or fewer samples.	Recovery ± 25% of true value, if sample > 4x spike value.	Flag results.
	Matrix Spike Duplicate Sample (P) or sample duplicate (D)	One per digestion batch of 20 or fewer samples.	Recovery ± 25% of true value, if sample < 4x spike added.	Flag results
	Post-Digestion Matrix Spike Sample (PDS)	When matrix spike or MSD fail	Recovery ±20% of true value	Analyze serial dilution of sample
	Serial Dilution Test (L)	One per digestion batch or when PDS fails	1:5 dilution of sample must agree within 10% with undiluted result	If MS, MSD, PDS, and serial dilution fail, quantitate sample by method of standard additions
	Instrument Detection Limit (IDL) Study	Quarterly.	IDL < PQL	1)Repeat IDL study. 2)Raise PQL.

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TABLE 1

QC REQUIREMENTS (CONTINUED)

Parameter/ Method	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Mercury/ USEPA Method 7471B	Method Detection Limit (MDL) Study Annually.	Refer to KAS SOP QA-806, "Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications", current revision.		
	Limit of Detection (LOD) determination	Quarterly.	LOD = 2-3X MDL	Repeat LOD Determination.

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TABLE 2

DoD QSM QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Demonstrate acceptable analytical capability	Prior to using any test method and at any time there is a significant change in instrument type, personnel, test method, or sample matrix.	QC acceptance criteria published by DoD, if available; otherwise, method-specified criteria.	Recalculate results; locate and fix problem, then rerun demonstration for those analytes that did not meet criteria.	NA.	This is a demonstration of analytical ability to generate acceptable precision and bias per the procedure in Appendix C. No analysis shall be allowed by analyst until successful demonstration of capability is complete.
LOD determination and verification	(Refer to current revision of SOP QA-806)				
LOQ establishment and verification	(Refer to current revision of SOP QA-806)				
Initial calibration (ICAL) for Mercury: minimum 5 standards and a calibration blank	Daily ICAL prior to sample analysis.	If more than one calibration standard is used, $r \geq 0.995$.	Correct problem, then repeat ICAL.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be run until ICAL has passed.
Second source calibration verification (ICV)	Once after each ICAL, prior to beginning a sample run.	Value of second source for all analyte(s) within ± 10% of true value.	Correct problem and verify second source standard. Rerun ICV. If that fails, correct problem and repeat ICAL.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be run until calibration has been verified.
Continuing calibration verification (CCV)	CVAA: within ± 20% of true value.	Correct problem, rerun calibration verification. If that fails, then repeat ICAL. Reanalyze all samples since the last successful calibration verification.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to all results for the specific analyte(s) in all samples since the last acceptable calibration verification.	After every 10 field samples and at the end of the analysis sequence.	Problem must be corrected. Results may not be reported without a valid CCV. Flagging is only appropriate in cases where the samples cannot be reanalyzed.

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TABLE 2

DoD QSM QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Method blank	One per preparatory batch.	No analytes detected > ½ RL and > 1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater). Blank result must not otherwise affect sample results. Contact Client if samples cannot be reprepped within hold time. For negative blanks, absolute value < LOD.	Correct the problem. Report sample results that are <LOD or >10x the blank concentration. Reprepare and reanalyze the method blank and all associated samples with results > LOD and < 10x the contaminated blank result. Contact Client if samples cannot be reprepped within hold time.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply B-flag to all results for the specific analyte(s) in all samples in the associated preparatory batch.	Problem must be corrected. Results may not be reported without a valid method blank. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Calibration blank	Before beginning a sample run, after every 10 samples, and at end of the analysis sequence.	No analytes detected > LOD. For negative blanks, absolute value < LOD.	Correct problem. Re-prep and reanalyze calibration blank. All samples following the last acceptable calibration blank must be reanalyzed.	Apply B-flag to all results for specific analyte(s) in all samples associated with the blank.	
LCS containing all analytes to be reported	One per preparatory batch.	Water: Recovery must be within ± 20% of the true value Soil: Recovery must be within vendor supplied limits (varies by lot).	Correct problem, then reprep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch.	Problem must be corrected. Results may not be reported without a valid LCS. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Matrix spike (MS)	One per preparatory batch per matrix (see Box D-7).	Recovery must be within ± 20% of the true value	Examine the project-specific DQOs. If the matrix spike falls outside of DoD criteria, additional quality control tests are required to evaluate matrix effects.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met.	For matrix evaluation only. If MS results are outside the LCS limits, the data shall be evaluated to determine the source of difference and to determine if there is a matrix effect or analytical error.
Matrix spike duplicate (MSD) or sample duplicate	One per preparatory batch per matrix	MSD: Recovery must be within ± 20% of the true value. MSD or sample duplicate: RPD ≤ 20% (between MS and MSD or sample and sample duplicate).	Examine the project-specific DQOs. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met.	The data shall be evaluated to determine the source of difference.

TABLE 2

DoD QSM QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Method of standard additions (MSA)	When matrix interference is confirmed.	NA.	NA.	NA.	Document use of MSA in the case narrative.
Results reported between DL and LOQ	NA.	NA.	NA.	Apply J-flag to all results between DL and LOQ.	

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TABLE 3

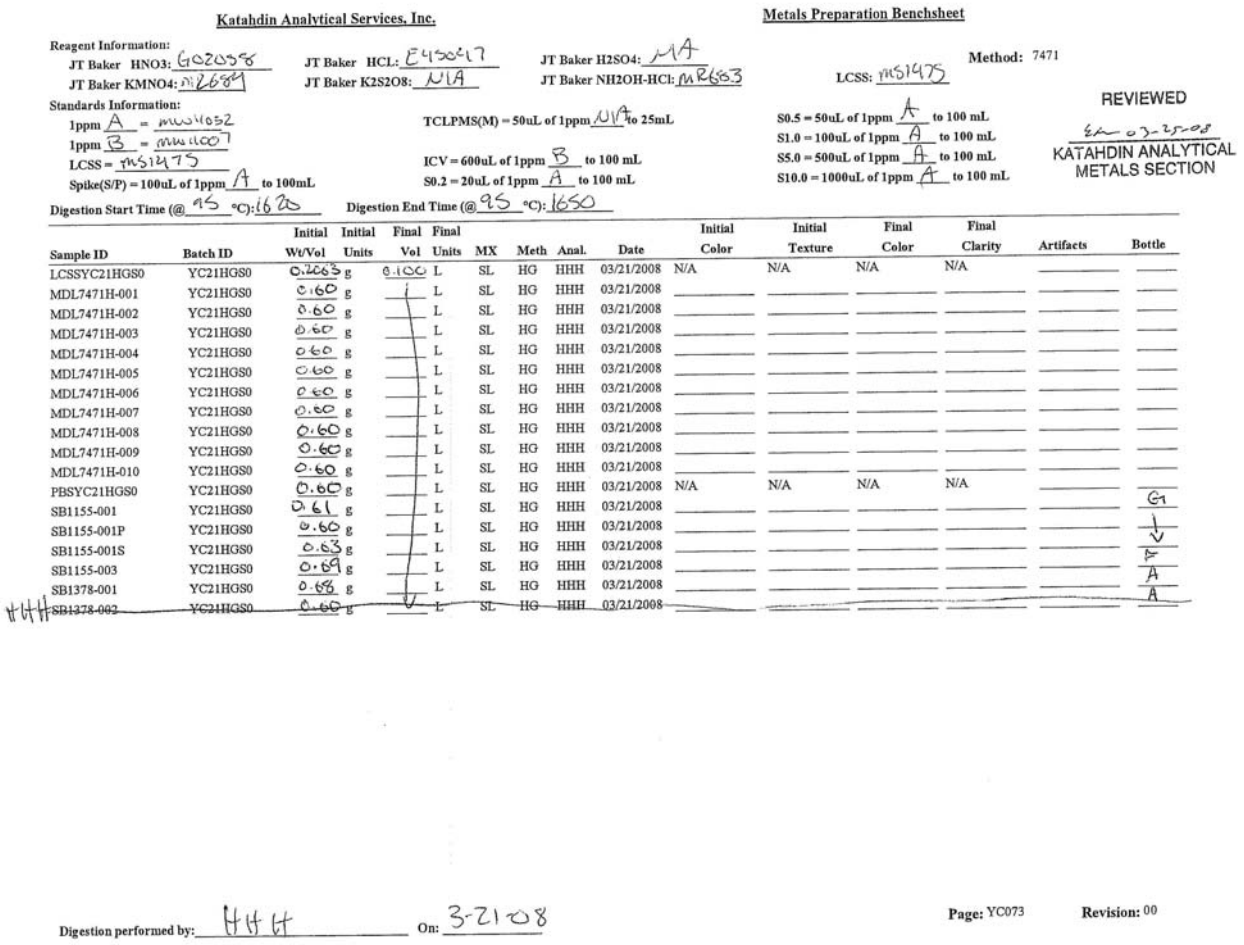
SUMMARY OF METHOD MODIFICATIONS

Topic	Katahdin SOP CA-611-07	USEPA Method 7471, current revision
Reagents	Stannous chloride dissolved in hydrochloric acid to prevent clogging of mercury analyzer, per instrument manufacturer's recommendation.	Stannous chloride dissolved/suspended in sulfuric acid.
Procedures	Sampling and gas stream switching performed automatically by mercury analyzer.	Sampling and gas stream switching performed manually by analyst.
QC – Calibration Verification	1)Known reference sample (ICV) analyzed daily. 2)Calibration verified after every 10 samples with CCV.	1)Known reference sample analyzed quarterly. 2)Calibration verified after every 20 samples.
QC - Calibration Blanks and Method Blanks	Acceptance Criterion: < PQL	Acceptance criteria: Low enough not to interfere with data quality objectives, or <10% of PQL, or <10% of regulatory limit, or <10% of lowest associated sample

TITLE: DIGESTION AND ANALYSIS OF SOLID SAMPLES FOR MERCURY BY USEPA METHOD 7471

FIGURE 1

EXAMPLE PAGE FROM MERCURY PREPARATION LOGBOOK



Katahdin Analytical Services, Inc.

Metals Preparation Benchsheet

Reagent Information:
JT Baker HNO3: G02058 JT Baker HCL: E43067 JT Baker H2SO4: NA
JT Baker KMNO4: N26891 JT Baker K2S2O8: N/A JT Baker NH2OH-HCl: MR6563
LCSS: MS1475 Method: 7471

Standards Information:
1ppm A = MW14052
1ppm B = MW14007
LCSS = MS1475
Spike(S/P) = 100uL of 1ppm A to 100mL
TCLPMS(M) = 50uL of 1ppm N/A to 25mL
ICV = 600uL of 1ppm B to 100 mL
S0.2 = 20uL of 1ppm A to 100 mL
S0.5 = 50uL of 1ppm A to 100 mL
S1.0 = 100uL of 1ppm A to 100 mL
S5.0 = 500uL of 1ppm A to 100 mL
S10.0 = 1000uL of 1ppm A to 100 mL

REVIEWED
EA 03-25-08
KATAHDIN ANALYTICAL METALS SECTION

Digestion Start Time (@ 95 °C): 1620 Digestion End Time (@ 95 °C): 1650

Sample ID	Batch ID	Initial Wt/Vol	Initial Units	Final Vol	Final Units	MX	Meth	Anal.	Date	Initial Color	Initial Texture	Final Color	Final Clarity	Artifacts	Bottle
LCSSYC21HGS0	YC21HGS0	0.2063	g	0.100	L	SL	HG	HHH	03/21/2008	N/A	N/A	N/A	N/A		
MDL7471H-001	YC21HGS0	0.60	g		L	SL	HG	HHH	03/21/2008						
MDL7471H-002	YC21HGS0	0.60	g		L	SL	HG	HHH	03/21/2008						
MDL7471H-003	YC21HGS0	0.60	g		L	SL	HG	HHH	03/21/2008						
MDL7471H-004	YC21HGS0	0.60	g		L	SL	HG	HHH	03/21/2008						
MDL7471H-005	YC21HGS0	0.60	g		L	SL	HG	HHH	03/21/2008						
MDL7471H-006	YC21HGS0	0.60	g		L	SL	HG	HHH	03/21/2008						
MDL7471H-007	YC21HGS0	0.60	g		L	SL	HG	HHH	03/21/2008						
MDL7471H-008	YC21HGS0	0.60	g		L	SL	HG	HHH	03/21/2008						
MDL7471H-009	YC21HGS0	0.60	g		L	SL	HG	HHH	03/21/2008						
MDL7471H-010	YC21HGS0	0.60	g		L	SL	HG	HHH	03/21/2008						
PBSYC21HGS0	YC21HGS0	0.60	g		L	SL	HG	HHH	03/21/2008	N/A	N/A	N/A	N/A		
SB1155-001	YC21HGS0	0.61	g		L	SL	HG	HHH	03/21/2008						G
SB1155-001P	YC21HGS0	0.60	g		L	SL	HG	HHH	03/21/2008						"
SB1155-001S	YC21HGS0	0.63	g		L	SL	HG	HHH	03/21/2008						"
SB1155-003	YC21HGS0	0.69	g		L	SL	HG	HHH	03/21/2008						A
SB1378-001	YC21HGS0	0.68	g		L	SL	HG	HHH	03/21/2008						A
HHH ~~SB1378-002~~	~~YC21HGS0~~	~~0.60~~	~~g~~		~~L~~	~~SL~~	~~HG~~	~~HHH~~	~~03/21/2008~~						

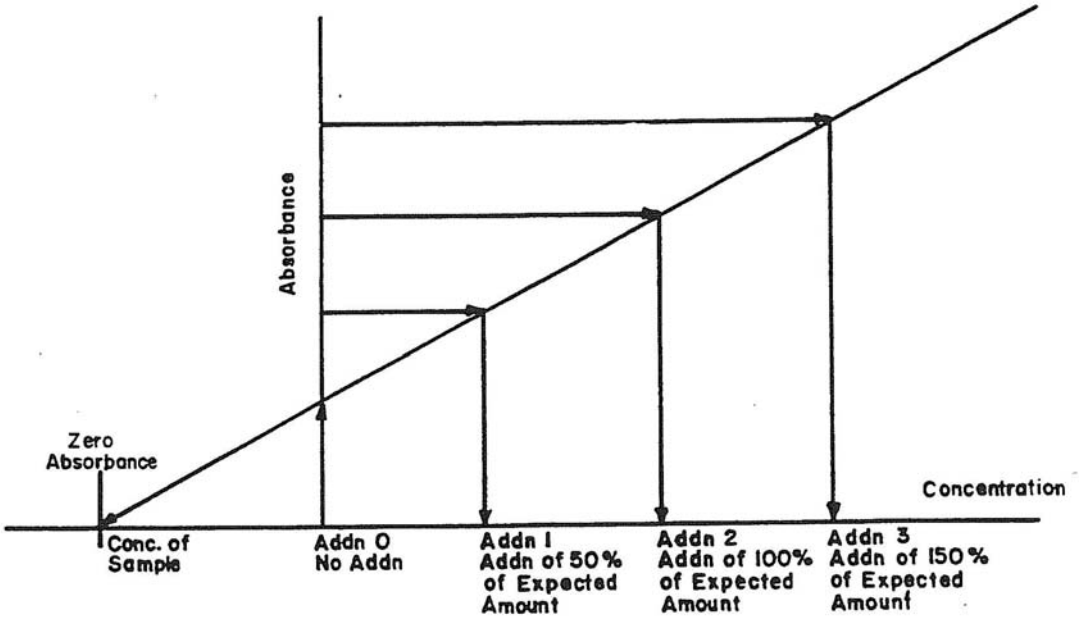
Digestion performed by: HHH On: 3-21-08

Page: YC073 Revision: 00

TITLE: DIGESTION AND ANALYSIS OF SOLID SAMPLES FOR MERCURY BY USEPA METHOD 7471

FIGURE 2

STANDARD ADDITIONS PLOT



TITLE: DIGESTION AND ANALYSIS OF SOLID SAMPLES FOR MERCURY BY USEPA METHOD 7471

FIGURE 3

EXAMPLE CERTIFICATE OF ANALYSIS FOR A SOLID REFERENCE MATERIAL

ENVIRONMENTAL RESOURCE ASSOCIATES®
The Industry Standard™

MS1475

DataPacK™
Lot No. D051-540

Trace Metals in Soil
Catalog No. 540

Certification

Method 3050 HNO3, H2O2, HCl Parameter	Total Concentration [1] (mg/Kg)	Certified Value [2] (mg/Kg)	Performance Acceptance Limits™ [3] (mg/Kg)
aluminum	55600*	7870	4630 - 11100
antimony	160	70.5	D.L. - 149
arsenic	316	289	234 - 344
barium	869	211	174 - 247
beryllium	60.9	54.4	45.2 - 63.6
boron	129	91.3	58.8 - 124
cadmium	114	101	82.9 - 119
calcium	9750*	3680	2970 - 4390
chromium	249	224	180 - 268
cobalt	113	101	82.7 - 119
copper	94.9	88.0	73.3 - 103
iron	24400*	15700	6610 - 24900
lead	184	158	129 - 187
magnesium	3780*	2260	1760 - 2750
manganese	703	420	343 - 497
mercury	5.32	5.18	3.42 - 6.87
molybdenum	80.2	69.6	55.5 - 83.7
nickel	137	120	99.1 - 141
potassium	33000*	3000	2200 - 3800
selenium	146	130	101 - 159
silver	127	104	68.9 - 139
sodium	15600*	1080	692 - 1470
strontium	326	113	90.5 - 135
thallium	106	94.0	72.8 - 115
tin	175	149	104 - 194
titanium	3100*	284	116 - 453
vanadium	151	111	85.1 - 137
zinc	311	272	215 - 329

Method 3050 HNO3, H2O2 Parameter	Total Concentration [1] mg/Kg	Certified Value [2] mg/Kg	Performance Acceptance Limits™ [3] mg/Kg
aluminum	55600*	7380	4440 - 10300
antimony	160	75.2	D.L. - 198
arsenic	316	284	225 - 343
barium	869	217	177 - 257
beryllium	60.9	53.6	42.7 - 64.5
boron	129	89.5	58.9 - 120
cadmium	114	103	83.6 - 122
calcium	9750*	3540	2800 - 4270
chromium	249	224	172 - 275
cobalt	113	101	82.0 - 120
copper	94.9	85.5	70.4 - 100
iron	24400*	12500	5480 - 19500
lead	184	162	132 - 192
magnesium	3780*	2160	1650 - 2670
manganese	703	415	330 - 500
mercury	5.32	5.18	3.42 - 6.87
molybdenum	80.2	68.8	52.7 - 84.9
nickel	137	119	98.5 - 140
potassium	33000*	2840	2160 - 3520
selenium	146	135	104 - 166
silver	127	107	49.8 - 164
sodium	15600*	1010	709 - 1310
strontium	326	111	89.0 - 133
thallium	106	99.3	76.8 - 122
tin	175	148	70.6 - 225
titanium	3100*	283	104 - 463
vanadium	151	104	70.5 - 138
zinc	311	275	222 - 328

KATAHDIN ANALYTICAL SERVICES, INC.
STANDARD OPERATING PROCEDURE

SOP Number: CA-615
Revision History
Cover Page
Page 1

TITLE: DIGESTION AND ANALYSIS OF AQUEOUS SAMPLES FOR MERCURY BY USEPA METHOD 7470

Prepared By: George Brewer Date: 01/01

Approved By:

Group Supervisor: George Brewer Date: 01/29/01

Operations Manager: Jol C. Burton Date: 1/29/01

QA Officer: Deborah J. Nadeau Date: 1·29·01

General Manager: Dennis P. Hogan Date: 1/29/01

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
00 7470A	NA	DH	1·29·01	1/29/01
01	Revised Sect. 4, 5 and 7 to reflect current practice. Revised Sect. 8 to reflect current QC limits. Revised sect. 10 to reflect current Applicable Documents and references. Removed figure 2. Update table 1 to reflect current QC limits. Minor changes throughout	LAD	02.16.05	02.16.05
02	Updated Fig. 1 - new prep logbook page	LAD	04/08	04/08
03	Updated Figure 1 - Example of a mercury Preparation Logbook page.	LAD	03/09	03/09
04	Added LOD definition. Updated sections 8, 9, 10 and Table 1 for DOD QSM version 4.1 compliance.	DH	08/09	08/09

TITLE: DIGESTION AND ANALYSIS OF AQUEOUS SAMPLES FOR MERCURY BY USEPA METHOD 7470

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
05	Added Table 2 - DoD QSM Version 4.1 QC Requirements	LAD	04/10	04/10

TITLE: DIGESTION AND ANALYSIS OF AQUEOUS SAMPLES FOR MERCURY BY USEPA METHOD 7470

Please acknowledge receipt of this standard operating procedure by signing and dating both of the spaces provided. Return the bottom half of this sheet to the QA Department.

I acknowledge receipt of copy ___ of document **SOP CA-615-05,** titled **DIGESTION AND ANALYSIS OF AQUEOUS SAMPLES FOR MERCURY BY USEPA METHOD 7470.**

Recipient: _____ Date: _____

KATAHDIN ANALYTICAL SERVICES, INC.
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy ___ of document **SOP CA-615-05,** titled **DIGESTION AND ANALYSIS OF AQUEOUS SAMPLES FOR MERCURY BY USEPA METHOD 7470.**

Recipient: _____ Date: _____

TITLE: DIGESTION AND ANALYSIS OF AQUEOUS SAMPLES FOR MERCURY BY USEPA METHOD 7470

1.0 SCOPE AND APPLICATION

The purpose of this SOP is to describe the procedure used by Katahdin Analytical Services, Inc. personnel for the digestion and analysis aqueous samples for mercury using cold vapor atomic absorption spectrophotometry.

This method is applicable to the determination of mercury in groundwaters, aqueous wastes, and mobility-procedure extracts under USEPA Method 7470 (Test Methods for Evaluating Solid Wastes: Physical/Chemical Methods, SW-846, 2nd edition, 1982 (revised 1984), 3rd edition, 1986, and Updates I, II, IIA, and III 1996, Office of Solid Waste and Emergency Response, U.S. EPA.

1.1 Definitions

CCB - Continuing Calibration Blank - An analyte-free solution consisting of acidified laboratory grade reagent water used to verify calibration accuracy periodically during analysis.

CCV - Continuing Calibration Verification - A midrange standard used to verify calibration accuracy periodically during analysis.

Duplicate - A second aliquot of a sample that is prepared and analyzed in the same way as the original sample in order to determine the precision of the method.

ICB - Initial Calibration Blank - An analyte-free solution consisting of acidified laboratory grade reagent water used to verify calibration accuracy.

ICV - Initial Calibration Verification - A standard made from a source independent from the calibration standards and with analyte concentrations different from those in the CCV; used to verify the accuracy of the instrument calibration.

IDL - Instrument Detection Limit - The lowest concentration of an analyte that can be determined with 95% confidence by the instrument.

LCS - Laboratory Control Sample - A standard or solid reference material that has been brought through the sample preparation process.

LOD – Limit of Detection – An estimate of the minimum amount of a substance that an analytical process can reliably detect. An LOD is analyte and matrix-specific and is used for DoD QSM acceptance criteria.

PB - Preparation Blank - Laboratory grade reagent water that has been brought through the sample preparation process.

PQL - Practical Quantitation Limit - The lowest concentration of an analyte that is routinely reported by the laboratory; nominally three to five times the IDL.

Matrix Spike - An aliquot of a sample to which a known amount of analyte has been added before digestion.

MDL - Method Detection Limit - The minimum concentration of an analyte that can be identified, measured, and reported with 99% confidence that the analyte concentration is greater than zero.

1.2 Responsibilities

This method is restricted to use by, or under the supervision of analysts experienced in the analysis of mercury by USEPA Method 7470. Each analyst must demonstrate and document their ability to generate acceptable results with this method. Refer to Katahdin SOP QA-805, current revision, “Personnel Training & Documentation of Capability”.

It is the responsibility of all Katahdin technical personnel involved in analysis of mercury by USEPA Method 7470 to read and understand this SOP, to adhere to the procedures outlined, and to properly document their data in the appropriate lab notebook. Any deviations from the test or irregularities with the samples should also be recorded in the lab notebook and reported to the Department Manager or designated qualified data reviewer responsible for this data.

It is the responsibility of the Department Manager to ensure that members of their group follow this SOP, that their work is properly documented, and to indicate periodic review of the associated logbooks.

1.3 Safety

Many of the samples and reagents used in cold vapor atomic absorption are toxic or corrosive. Rubber gloves, safety glasses, lab coats, and other protective clothing should be worn whenever these materials are handled. Because of the toxic nature of mercury vapor, care must be taken to avoid its inhalation. The instrument exhaust fan must be in operation whenever the mercury analyzer is in use (the fan should never be shut off).

Users of this procedure must be cognizant of inherent laboratory hazards, proper disposal procedures for contaminated materials and appropriate segregation of hazardous wastes. The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical should be treated as a potential health hazard. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Everyone involved with

the procedure must be familiar with the MSDSs for all the materials used in this procedure.

Each qualified analyst or technician must be familiar with the Katahdin Analytical Environmental Health and Safety Manual including the Katahdin Hazardous Waste Management Plan and follow appropriate procedures such as wearing safety glasses and gloves when working with chemicals or near an instrument and not taking food or drink into the laboratory. Each analyst should know the location and use of all safety equipment.

1.4 Pollution Prevention/Waste Disposal

Whenever possible, laboratory personnel should use pollution prevention techniques to address their waste generation. Refer to the current revision of the Katahdin Hazardous Waste Management Plan for further details on pollution prevention techniques.

Samples, sample digestates, standards, and other reagents used in cold vapor atomic absorption may contain high concentrations of acids, mercury, and other toxic metals. They should be disposed of in a manner appropriate to the types of hazards they present. All digested mercury samples and standards and excess reagents and standards should be disposed of in the satellite waste container for corrosive wastes (labeled “Waste Stream A”) that is located in the Metals Prep lab. Further information regarding waste classification and disposal may be obtained by consulting the laboratory's Hazardous Waste Management Plan and Safety Manual and the Department Manager.

2.0 SUMMARY OF METHOD

The cold vapor atomic absorption technique is based on the absorption of radiation at 253.7 nm by mercury vapor. It relies on the volatility of elemental mercury at room temperature. During preparation, organic mercurials are oxidized and elemental mercury is ionized to Hg^{3+}. During instrumental analysis, mercuric ions are reduced to elemental mercury by the addition of stannous chloride. Elemental mercury is then aerated from solution and passes through a cell positioned in the path of a mercury spectrophotometer, where absorbance (peak height) is measured as a function of mercury concentration and recorded by the associated computer. The mercury vapor is then swept out of the instrument into an exhaust hood, where it is evacuated from the laboratory.

3.0 INTERFERENCES

In addition to inorganic forms of mercury, organic mercurials may be present in environmental samples. These organo-mercury compounds will not respond to the cold

vapor atomic absorption technique unless they are first broken down and converted to mercuric ions. The presence of undigested organo-mercurials in samples will result in a low bias for analytical results. Certain volatile organic materials will also non-specifically absorb radiation at the 253.7 nm analytical wavelength. The presence of such compounds may result in a high bias for analytical results. For these reasons, complete digestion using potassium permanganate and potassium persulfate is required for all environmental samples. Complete digestion is indicated by the persistence of the purple permanganate color (indicating the presence of excess permanganate) following digestion.

Sea waters, brines, and industrial effluents high in chlorides may require additional permanganate to maintain a persistent purple color following digestion. During the oxidation step, chlorides are converted to free chlorine which will absorb radiation at the 253.7 nm analytical wavelength. Any free chlorine thus generated will be present in the headspace of the digestion vessel following digestion. Because samples are poured into autosampler tubes prior to analysis by the mercury analyzer, any free chlorine present in the headspace of the digestion vessels is not sampled by the instrument and the analysis is free of chlorine interference.

4.0 APPARATUS AND MATERIALS

4.1 40 mL VOA vials, for use as digestion vessels.

4.2 250 mL Pyrex media bottles with plastic screw caps, for use in digesting calibration standards.

4.3 Water bath capable of maintaining a constant temperature of 95^{o} C.

4.4 Adjustable volume automatic pipettes - 2 to 20 uL, 10 to 100 uL, 100 to 1000 uL. Calibrated Eppendorf Reference pipets and Finn digital pipets are appropriate.

4.5 Repipetters (adjustable repeating pipetters with reservoirs) for dispensing concentrated nitric acid, concentrated sulfuric acid, and other reagents

4.6 Spirit-filled thermometer, NIST-traceable, covering the range from 20^{o} to 110^{o} C, for monitoring the temperature of the water bath. Mercury-filled thermometers are not acceptable for use in the metals laboratory, due to the possibility of breakage and consequent contamination.

4.7 Disposable graduated polystyrene sample cups, 200 mL capacity

4.8 CETAC M-6100 automated mercury analyzer and associated peripherals and parts

4.9 Disposable graduated dose cups, 30 mL capacity

TITLE: DIGESTION AND ANALYSIS OF AQUEOUS SAMPLES FOR MERCURY BY USEPA METHOD 7470

Refer to Katahdin SOP CA-629, current revision, "Operation and Maintenance of the CETAC M-6100 Automated Mercury Analyzer" for additional required materials.

5.0 REAGENTS

5.1 Laboratory grade reagent water – mercury-free water meeting the specifications of ASTM Type II water

5.2 Concentrated sulfuric acid, trace metals grade

5.3 Concentrated nitric acid, trace metals grade

5.4 Concentrated hydrochloric acid, trace metal grade

5.5 Potassium permanganate solution, 5% w/v: Dissolve 50 g of potassium permanganate in 1 L laboratory grade reagent water. The source reagent should be labeled as suitable for use in mercury determination.

5.6 Potassium persulfate solution, 5% w/v: Dissolve 50g of potassium permanganate in 1L laboratory grade reagent water. The source reagent should be labeled as suitable for use in mercury determination.

5.7 Sodium chloride – hydroxylamine hydrochloride solution: Dissolve 120 g sodium chloride and 120 g hydroxylamine hydrochloride in laboratory grade reagent water and dilute to a final volume of 1 L.

5.8 Stannous chloride solution: Add 70 mL concentrated hydrochloric acid to 500 mL of laboratory grade reagent water. Add 100 g stannous chloride and bring to a final volume of 1 L. Mix to dissolve. Reagent should be labeled as suitable for use in mercury determination.

5.9 Intermediate Mercury Standard A: Appropriately dilute a mercury stock standard to obtain a solution containing 1000 ug of mercury per liter in 2% nitric acid. This intermediate standard is used to prepare calibration standards, matrix spikes, CCVs, and laboratory control samples (refer to Section 8). The identity of the stock standard currently used to prepare this intermediate may be obtained by consulting the Standards Preparation Logbook maintained in the Section. Intermediate Mercury Standard A must be prepared fresh monthly ,and disposed of appropriately after use. (Note: the concentrations of all stock standards must be certified by the vendors as traceable to NIST reference materials).

5.10 Intermediate Mercury Standard B: Appropriately dilute a mercury stock standard to obtain a solution containing 1000 ug of mercury per liter in 2% nitric acid. The source of the stock standard used to prepare Intermediate Mercury Standard B must

be distinct from that used to prepare Intermediate Mercury Standard A (i.e. obtained from a separate vendor). Intermediate Mercury Standard B is used to prepare the ICV (refer to Section 8). The identity of the stock standard currently used to prepare this intermediate standard may be obtained by consulting the Standards Preparation Logbook maintained in the Section. Intermediate Mercury Standard B must be prepared fresh monthly, and disposed of appropriately after use.

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

Aqueous samples to be analyzed for mercury should be collected and preserved as described in the following table.

Matrix	Container[1]	Collection Volume/ Weight	Preservation/ Treatment	Holding Time
Aqueous (total)	P, G	250 mL	HNO_3 to pH < 2	28 days
Aqueous (dissolved)	P, G	250 mL	HNO_3 to pH < 2	28 days

[1] P = polyethylene or G = glass

7.0 PROCEDURES

BOTTLE PREPARATION

7.1 Mercury digestions are performed in two different types of vessels. Calibration standards, the Initial Calibration Verification (ICV) standard, and the Initial/Continuing Calibration Blank (ICB/CCB) are prepared in 250 mL Pyrex media bottles. Large bottles are used to provide sufficient volumes of these standards to allow for multiple reanalyses when required. Field samples, Method Blanks, and Laboratory Control Samples are digested in 40 mL VOA vials. These smaller vials provide enough digestate to allow one or two reanalyses when required, but reduce the amounts of samples consumed and waste generated.

VOA vials are reused if the samples they have contained have no measurable mercury above the PQL. After the previous contents of the vials have been discarded, these vials are segregated according to whether the measured mercury concentrations of the previous contents were above the PQL (contaminated vials) or below the PQL (uncontaminated vials). Labels are removed from the vials by wiping with a paper towel saturated with toluene. Uncontaminated vials are rinsed with laboratory grade reagent water. Contaminated vials are discarded.

The Pyrex media bottles in which standards are prepared are emptied, rinsed, and reused. Each of these bottles is permanently marked with the concentration of the standard it contains.

PREPARATION OF STANDARDS, QC SAMPLES, AND BLANKS

7.2 Prior to performing the digestion, make a list of the samples that are to be digested. Enter digestion information (Katahdin Sample Numbers, QC Batch ID, preparation date, analyst initials, etc.) into the ACCESS Metals database and print out a copy of the sample prep bench sheet. All necessary details of sample preparation (standards preparation information, digestion times, initial and final volumes, pertinent observations, etc.) must be recorded on this spreadsheet, which will be bound in the Mercury Preparation Logbook. Refer to Figure 1 for an example page from the Mercury Preparation Logbook.

7.3 Using a silver paint marker, label clean VOA vials with the appropriate sample numbers and standard identifications for each sample and standard to be digested.

7.4 Use a bottle-top dispenser to add 100 mL of laboratory grade reagent water to 6 standards digestion bottles (250 mL media bottles). Using calibrated adjustable pipettes, prepare calibration standards by adding 0 uL, 20 uL, 50 uL, 100 uL, 500 uL, and 1000 uL of Intermediate Mercury Standard A to separate appropriately-labeled media bottles containing 100 mL of laboratory grade reagent water. The mercury concentrations of these calibration standards are, respectively, 0 ug/L (calibration blank), 0.2 ug/L, 0.5 ug/L, 1.0 ug/L, 5.0 ug/L, and 10.0 ug/L. The 0.2 ug/L and 0.5 ug/L standards are analyzed after calibration as the PQL standard and the CCV (refer to Section 8.0), respectively, as well as being used in the creation of the calibration curve.

7.5 Add 100 mL of laboratory grade reagent water to the media bottle labeled “ICV”. Using a calibrated adjustable pipette, prepare the Initial Calibration Verification standard (refer to Section 8) by adding 600 uL of Intermediate Mercury Standard B to the water in this bottle, and record the bottle number in the Mercury Preparation Logbook. The mercury concentration of the ICV is 6.0 ug/L.

7.6 Prepare an appropriate number of preparation blanks (PBW) by adding 25 mL of laboratory grade reagent water to labeled vials.

7.7 Prepare an appropriate number of laboratory control samples (LCSW) by adding 125 uL of Intermediate Mercury Standard A to labeled digestion vials containing 25 mL of laboratory grade reagent water. The mercury concentration of each LCSW is 5.0 ug/L.

7.8 Matrix spikes are prepared by adding 25 uL of Intermediate Mercury Std A to 25 mL aliquots of samples. The concentration of mercury added to each matrix spike is 1.0 ug/L.

7.9 All QC samples and blanks are digested in the same manner as client samples. Refer to Sample Preparation and Digestion, sections 7.10 through 7.13 of this SOP. The volumes of reagents added to the standards prepared in the media bottles are four times those listed in sections 7.10 through 7.13.

SAMPLE PREPARATION AND DIGESTION

7.10 Using a graduated disposable dosecup, transfer 25 mL of sample, or an aliquot diluted to 25 mL, to a digestion vial. Add 1.25 mL of concentrated sulfuric acid and 0.625 mL of concentrated nitric acid, swirling to mix after each addition. Add 3.75 mL of potassium permanganate solution, swirl to mix, and allow to stand for at least 15 minutes. Samples that contain large amounts of organic substances may require additional 3.75 mL aliquots of potassium permanganate solution. This is indicated by the failure of the purple permanganate color to persist for the entire 15 minute waiting period. Add additional 3.75 mL aliquots to samples as necessary until the purple color persists for 15 minutes. If any of the samples require these additional aliquots of potassium permanganate solution, record the additional volume used for each sample on the mercury preparation benchsheet.

7.11 Add 2 mL of potassium persulfate solution to each sample. Cap the vials and place them in a preheated water bath. Monitor the temperature of the bath with a spirit thermometer throughout the digestion. The temperature of the water bath will fall below 95^{o} C upon addition of the digestion vials. After the temperature of the bath has risen back to 95^{o} C, continue heating the samples at 95^{o} C for two hours. Record initial and final digestion times and temperatures in the mercury prepareation benchsheet.

7.12 Remove bottles from the water bath and allow to cool to room temperature. If the purple permanganate color has failed to persist after digestion in any of the samples, add additional 3.75 mL aliquots of potassium permanganate solution as required to the samples, and record these additions in the mercury preparation benchsheet. Heat the samples that required additional permanganate in the water bath at 95^{o} C for an additional two hours. Remove the bottles from the water bath and allow to cool to room temperature. If the purple color fails to persist after the second heating step, consult the Department Manager for advice on how to proceed.

7.13 Add 1.5 mL of sodium chloride – hydroxylamine hydrochloride solution to each digestion vial and swirl to mix. This will reduce the excess permanganate, and the sample will change from purple to colorless. Wait at least 30 seconds before proceeding with analysis.

INSTRUMENTAL ANALYSIS

7.14 Digested mercury samples are analyzed using the CETAC M-6100 Automated Mercury Analyzer. Analysis is automated and is controlled by the QuickTrace Mercury Analyzer software running on a dedicated PC. Detailed instructions for setting up the instrument and analyzing samples are given Katahdin SOP CA-629, "Operation and Maintenance of the CETAC M-6100 Automated Mercury Analyzer".

METHOD OF STANDARD ADDITIONS

7.15 The standard addition technique involves adding known amounts of standard to one or more aliquots of the processed sample solution. This technique compensates for a sample constituent that enhances or depresses the analyte signal, thus producing a different slope from that of the calibration standards. It will not correct for additive interferences which cause a baseline shift. The method of standard additions shall be used for analysis of all EP extracts, on all analyses submitted as part of a delisting petition, and whenever a new sample matrix is being analyzed.

7.15.1 The simplest version of this technique is the single-addition method, in which two identical aliquots of the sample solution, each of volume V_x, are taken. To the first (labeled A) is added a known volume V_S of a standard analyte solution of concentration C_S. To the second aliquot (labeled B) is added the same volume V_S of the solvent. The analytical signals of A and B are measured and corrected for nonanalyte signals. The unknown sample concentration C_x is calculated:

$$C_X = \frac{S_B V_S C_S}{(S_A - S_B)V_X}$$

where S_A and S_B are the analytical signals (corrected for the blank) of solutions A and B, respectively. V_s and C_s should be chosen so that S_A is roughly twice S_B on the average, avoiding excess dilution of the sample. If a separation or concentration step is used, the additions are best made first and carried through the entire procedure.

7.15.2 Improved results can be obtained by employing a series of standard additions. To equal volumes of the sample are added a series of standard solutions containing different known quantities of the analyte, and all solutions are diluted to the same final volume. For example, addition 1 should be prepared so that the resulting concentration is approximately 50 percent of the expected absorbance from the endogenous analyte in the sample. Additions 2 and 3 should be prepared so that the concentrations are approximately 100 and 150 percent of the expected endogenous sample absorbance. The absorbance of each solution is determined and then plotted on the vertical axis of a graph, with the concentrations of the known

standards plotted on the horizontal axis. When the resulting line is extrapolated to zero absorbance, the point of interception of the abscissa is the endogenous concentration of the analyte in the sample. The abscissa on the left of the ordinate is scaled the same as on the right side, but in the opposite direction from the ordinate. An example of a plot so obtained is shown in Figure 3. A linear regression program may be used to obtain the intercept concentration.

7.15.3 For the results of this MSA technique to be valid, the following limitations must be taken into consideration:

- The apparent concentrations from the calibration curve must be linear over the concentration range of concern. For the best results, the slope of the MSA plot should be nearly the same as the slope of the standard curve. If the slope is significantly different (greater than 20%), caution should be exercised.

- The effect of the interference should not vary as the ratio of analyte concentration to sample matrix changes, and the standard addition should respond in a similar manner as the analyte.

- The determination must be free of spectral interference and corrected for nonspecific background interference.

DATA REDUCTION AND REPORTING

7.16 Results are obtained in concentration units (ug/L) from the instrument. Electronic instrument data files are imported into the Metals ACCESS database for data reduction. Sample preparation information (initial sample volumes and final digestate volumes) are entered directly into the Metals ACCESS database to allow calculation of final results for reporting. Results are calculated as follows:

$$\text{Mercury concentration (ug/L)} = \frac{MC \times DF \times IV}{FV}$$

Where: MC = Measured mercury concentration (ug/L)
DF = Dilution factor at instrument
IV = Initial sample volume (mL)
FV = Final digestate volume (mL)

7.17 Results that exceed the calibration range of the instrument may not be reported - the sample must be appropriately diluted and reanalyzed. Results for diluted samples should be multiplied by the dilution factor prior to reporting. If additional aliquots of potassium permanganate were added during digestion, the resulting dilution must be corrected for before reporting.

7.18 Results are reported down to the laboratory's practical quantitation level (PQL), unless otherwise requested. Results below the PQL should be reported to the PQL and flagged with a "U" qualifier.

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

USEPA Method 7470 requires the laboratory to perform specific quality control checks to assess laboratory performance and data quality. Minimum frequencies, acceptance criteria, and corrective actions for these control checks are tabulated in Table 1 and are described below. Preparation instructions and the resulting mercury concentrations for calibration standards, QC standards, and matrix spikes are detailed in Sections 7.4 through 7.8 of this SOP. Table 1 criteria are intended to be guidelines for analysts. The table does not cover all possible situations. If any of the QC requirements are outside the recovery ranges listed in Table 1, all associated samples must be evaluated against all the QC. In some cases data may be reported, but may be reanalyzed in other cases. Making new reagents and standards may be necessary if the standardization is suspect. The corrective actions listed in Table 1 may rely on analyst experience to make sound scientific judgments. These decisions are based on holding time considerations and client and project specific Data Quality Objectives. The Department Manager, Operations Manager, General Manager and/or Quality Assurance Officer may be consulted to evaluate data. Some samples may not be able to be reanalyzed within hold time. In these cases "qualified" data with narration may be advisable after consultation with the client.

In some cases the standard QC requirements listed in Table 1 may not be sufficient to meet the Data Quality Objectives of the specific project. Much of the work performed at the lab is analyzed in accordance with specific QC requirements spelled out in a project specific Quality Assurance Project Plan (QAPP) or in a program specific Quality Systems Manual (QSM). The reporting limits, acceptance criteria and/or corrective actions may be different than those specified in this SOP. In these cases the appropriate information will be communicated to the Department Manager and/or senior chemists before initiation of the analyses so that specific product codes can be produced for the project. In addition, the work order notes for each project will describe the specific QAPP or QSM to be followed.

INITIAL DEMONSTRATION OF PERFORMANCE

8.1 Instrument detection limits (IDL) are determined quarterly for each analyte analyzed on each instrument by each method. This determination requires seven replicate analyses of a laboratory grade reagent water spiked at 3-5 times the anticipated detection limit for each analyte, performed on three non-consecutive days. The standard deviation of the 21 analyses is multiplied by three to obtain the IDL. For more information on performing IDL determinations, refer to the current revision of Katahdin SOP QA-806.

8.2 Method detection limits (MDL) are determined annually for each analyte analyzed on each instrument. This determination requires at least seven replicate digestions

and analyses of laboratory grade reagent water spiked at 3-5 times the anticipated MDL for each analyte. MDLs differ from IDLs in that the replicates are digested prior to analysis, and they may be analyzed on a single day. The standard deviation of the 7 (or more) replicate analyses is multiplied by the Student's t-value to obtain the MDL. For more information on performing MDL determinations, refer to the current revision of Katahdin SOP QA-806.

8.3 Limits of Detection (LOD) are used when evaluating data using DoD QSM. The LOD is established by spiking a quality system matrix at 2-3 times the detection limit for a single analyte standard and 1-4 times the detection limit for a multi-analyte standard. The LOD must be verified quarterly. For more information on performing LOD determinations, refer to the current revision of Katahdin SOP QA-806.

8.4 Instrument calibration - The instrument must be calibrated each time it is set up, and calibration standards must be digested each day that samples are digested. Calibration includes analysis of a calibration blank and five calibration standards with graduated concentrations in the appropriate range. The concentration of one of the calibration standards must be at the Practical Quantitation Level (PQL). The intermediate standards used for preparing the calibration standards are prepared at least once per month in 2% nitric acid. Because mercury may be adsorbed onto the walls of glass and plastic containers, the calibration standards must be prepared fresh daily. The correlation coefficient for the calibration curve must be at least 0.995. If the calibration curve does not pass this test, analysis must be halted, the problem corrected, and the instrument recalibrated.

8.5 An Initial Calibration Verification (ICV) solution is analyzed after the initial calibration to check calibration accuracy. The ICV solution is prepared from a standard source different than that of the calibration standard and at a concentration within the working range of the instrument. The result of the ICV must fall within 90% to 110% of the expected value. If the ICV fails, results may not be reported from the run until the problem is corrected and a passing ICV has been analyzed.

8.6 The Continuing Calibration Verification (CCV) solution is analyzed after the initial calibration, after every ten samples, and at the end of the analytical run. The CCV solution is prepared using the same standard used for calibration at a concentration near the mid-point of the calibration curve. Results of the CCVs must fall within 80% to 120% of the expected value. If a CCV fails, associated sample results may not be reported from the run until the problem is corrected and a passing CCV has been analyzed. Also, all samples analyzed after the last passing CCV must be reanalyzed.

8.7 A calibration blank is analyzed after each ICV and CCV. A calibration blank that is analyzed after the ICV is called an Initial Calibration Blank (ICB). A calibration blank that is analyzed after a CCV is called a Continuing Calibration Blank (CCB). The absolute values of results of ICBs and CCBs must be less than the Practical

Quantitation Level (PQL) for each element. If samples are being run using DoD QSM criteria, the absolute values of ICBs and CCBs must be less than the Limit of Detection (LOD). If an ICB or a CCB fails, results for the failing elements may not be reported from the run until the problem is corrected and a passing ICB or CCB has been analyzed. Also, all samples analyzed after the last passing CCB must be reanalyzed.

8.8 A standard with a mercury concentration that is at the Practical Quantitation Limit (PQL) is analyzed at the beginning of the run to determine calibration accuracy at the reporting limit. Result of the PQL standard should fall within 70% to 130% of the expected values. No corrective action has been established at this time.

PREPARATION BATCH QC SAMPLES

8.9 Preparation blank (PBW or PBS), consisting of reagent water carried through the same process as associated samples, is prepared with each digestion batch of twenty or fewer samples. The results of preparation blanks must be less than the Practical Quantitation Level (PQL) for each element. For DoD QSM acceptance criteria the results must be less than ½ the PQL except for common contaminants which must be less than the PQL. If a preparation blank fails, results for the failing elements may not be reported from the digestion batch, and all associated samples must be redigested, with the following exception. If the result for a preparation blank is greater than the PQL (greater than ½ PQL for DoD), associated sample results that are less than the PQL (less than ½ PQL for DoD) or greater than or equal to ten times the measured preparation blank concentration may be reported.

8.10 A laboratory control sample (LCSW), consisting of spiked reagent carried through the same process as associated samples, is prepared with each digestion batch of twenty or fewer samples. Results for laboratory control samples must fall within 80% to 120% of the expected value, unless laboratory-generated statistical limits are available. If a laboratory control sample fails, results may not be reported from the digestion batch, and all associated samples must be redigested.

SAMPLE MATRIX QC SAMPLES

8.11 Matrix spiked duplicate samples are prepared at a minimum frequency of one per digestion batch. Matrix spike recoveries for these samples are calculated as follows:

$$\text{Recovery (\%)} = \frac{(P - S)}{A} \times 100\%$$

where: P = Spiked sample value
S = Original sample value
A = Spike amount

The recovery for each element in a spiked sample or spiked duplicate sample must fall within 75% to 125% of the actual value if the result for the unspiked sample is less than four times the amount of spike added. If one or both spike recoveries fail, a matrix interference should be suspected and the associated sample result should be flagged on the report of analysis. If DoD QSM acceptance criteria are being used, recoveries must be the same as stated for laboratory control samples.

The relative percent difference between matrix spiked duplicate sample results is calculated as follows:

$$RPD\ (\%) = \frac{|D_1 - D_2|}{(|D_1 + D_2|)/2} \times 100$$

where: D_1 = Spike sample result
D_2= Spike duplicate sample result

A control limit of 20% RPD is applied to matrix spike duplicate analysis. If the matrix spike duplicate analysis fails, the associated sample result should be flagged on the report of analysis.

9.0 METHOD PERFORMANCE

The method detection limit (MDL) and the limit of detection (LOD) are defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero. The MDLs and LODs are determined prior to sample analysis per type of instrument and filed with the Inorganic Department Manager and with the QAO. Refer to the current revision of Katahdin SOP QA-806, Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications, for procedures on determining the MDL.

Refer to the current revision of USEPA Method 7470 for other method performance parameters and requirements.

10.0 APPLICABLE DOCUMENTS/REFERENCES

Department of Defense Quality Systems Manual for Environmental Laboratories (DOD QSM), Version 4.1, 04/22/09.

Katahdin SOP CA-101, Equipment Maintenance, current revision.

Katahdin SOP QA-806, Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications.

TITLE: DIGESTION AND ANALYSIS OF AQUEOUS SAMPLES FOR MERCURY BY USEPA METHOD 7470

The National Environmental Laboratory Accreditation Conference (NELAC) Standards, June 2003.

Test Methods for Evaluating Solid Wastes, United States Environmental Protection Agency, USEPA SW 846, Third Edition, Final Update III (9/94), Method 7470A.

QuickTrace M6100 Mercury Analyzer Operator Manual Version 1.0.1, CETAC Technologies.

QuickTrace Mercury Analyzer Software Manual, CETAC Technologies.

List of Tables and Figures

TITLE: DIGESTION AND ANALYSIS OF AQUEOUS SAMPLES FOR MERCURY BY USEPA METHOD 7470

TABLE 1

QC REQUIREMENTS

Parameter/ Method	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Mercury/ USEPA 7470	Initial Calibration, 5 points plus a calibration blank.	Daily prior to sample analysis.	Correlation coefficient ≥ 0.995.	Correct problem and repeat calibration.
	Initial Calibration Verification (ICV), prepared from a second source.	Before beginning a sample run.	Recovery within ± 10% of true value.	Correct problem and repeat calibration.
	Initial Calibration Blank (ICB)	Before beginning a sample run.	Less than PQL.	Correct problem and repeat calibration.
	Practical Quantitation Level Standard (PQL)	Before beginning a sample run.	Recovery within ± 30% of true value.	No corrective action required at this time.
	Continuing Calibration Verification (CCV)	At beginning or run, after every 10 samples, and at end of the run	Recovery within ± 20% of true value	Repeat calibration and reanalyze all samples analyzed since the last successful CCV.
	Continuing Calibration Blank (CCB)	At beginning or run, after every 10 samples, and at end of the run	Less than PQL.	Repeat calibration and reanalyze all samples analyzed since the last successful CCB.
	Preparation Blank (PBW)	One per digestion batch of 20 or fewer samples.	Less than PQL.	1) Investigate source of contamination. 2) Redigest and reanalyze all associated samples if sample concentration ≥ PQL and < 10x the blank concentration.
	Laboratory Control Sample (LCSW)	One per digestion batch of 20 or fewer samples.	Recovery within ± 20% of true value.	Redigest all affected samples.
	Matrix Spike Sample (S)	One per digestion batch of 20 or fewer samples.	Recovery ±25% of true value, if sample > 4x spike value.	Flag results.
	Matrix Spike Duplicate Sample (P)	One per digestion batch of 20 or fewer samples.	1) Recovery ± 25% of true value, if sample < 4x spike added. 2) RPD ≤20% for duplicate spikes.	Flag results
	Instrument Detection Limit (IDL) Study	Quarterly.	IDL < PQL	1) Repeat IDL study. 2) Raise PQL.
	Limit of Detection (LOD) determination	Quarterly.	LOD = 2-3X MDL	Repeat LOD Determination.
	Method Detection Limit (MDL) Study	Refer to KAS SOP QA-806, "Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications", current revision.		

TABLE 2

DOD QSM VERSION 4.1 QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Demonstrate acceptable analytical capability	Prior to using any test method and at any time there is a significant change in instrument type, personnel, test method, or sample matrix.	QC acceptance criteria published by DoD, if available; otherwise, method-specified criteria.	Recalculate results; locate and fix problem, then rerun demonstration for those analytes that did not meet criteria.	NA.	This is a demonstration of analytical ability to generate acceptable precision and bias per the procedure in Appendix C. No analysis shall be allowed by analyst until successful demonstration of capability is complete.
LOD determination and verification	(Refer to current revision of SOP QA-806)				
LOQ establishment and verification	(Refer to current revision of SOP QA-806)				
Initial calibration (ICAL) for mercury - minimum 5 standards and a calibration blank	Daily ICAL prior to sample analysis.	5 points plus a calibration blank, $r \geq 0.995$.	Correct problem, then repeat ICAL.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be run until ICAL has passed.
Second source calibration verification (ICV)	Once after each ICAL, prior to beginning a sample run.	Value of second source for all analyte(s) within ± 10% of true value.	Correct problem and verify second source standard. Rerun ICV. If that fails, correct problem and repeat ICAL.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be run until calibration has been verified.
Continuing calibration verification (CCV)	within ± 20% of true value.	Correct problem, rerun calibration verification. If that fails, then repeat ICAL. Reanalyze all samples since the last successful calibration verification.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to all results for the specific analyte(s) in all samples since the last acceptable calibration verification.	After every 10 field samples and at the end of the analysis sequence.	Problem must be corrected. Results may not be reported without a valid CCV. Flagging is only appropriate in cases where the samples cannot be reanalyzed.

TITLE: DIGESTION AND ANALYSIS OF AQUEOUS SAMPLES FOR MERCURY BY USEPA METHOD 7470

TABLE 2

DOD QSM VERSION 4.1 QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Method blank	One per preparatory batch.	No analytes detected > ½ RL (> RL for common lab contaminants) and > 1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater). Blank result must not otherwise affect sample results.	Correct the problem. Report sample results that are <LOD or >10x the blank concentration. Reprepare and reanalyze the method blank and all associated samples with results > LOD and < 10x the contaminated blank result. Contact Client if samples cannot be reprepped within hold time.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply B-flag to all results for the specific analyte(s) in all samples in the associated preparatory batch.	Problem must be corrected. Results may not be reported without a valid method blank. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Calibration blank	Before beginning a sample run, after every 10 samples, and at end of the analysis sequence.	No analytes detected > LOD.	Correct problem. Re-prep and reanalyze calibration blank. All samples following the last acceptable calibration blank must be reanalyzed.	Apply B-flag to all results for specific analyte(s) in all samples associated with the blank.	
LCS	One per preparatory batch.	Water: Recovery must be within + 20% of the true value Soil: Recovery must be within vendor supplied limits (varies by lot).	Correct problem, then reprep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available (see full explanation in Appendix G).	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch.	Problem must be corrected. Results may not be reported without a valid LCS. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Matrix spike (MS)	One per preparatory batch per matrix (see Box D-7).	Recovery must be within + 20% of the true value.	Examine the project-specific DQOs. If the matrix spike falls outside of DoD criteria, additional quality control tests are required to evaluate matrix effects.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met.	For matrix evaluation only. If MS results are outside the LCS limits, the data shall be evaluated to determine the source of difference and to determine if there is a matrix effect or analytical error.
Matrix spike duplicate (MSD) or sample duplicate	One per preparatory batch per matrix (see Box D-7).	MSD: Recovery must be within + 20% of the true value. MSD or sample duplicate: RPD ≤ 20% (between MS and MSD or sample and sample duplicate).	Examine the project-specific DQOs. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met.	The data shall be evaluated to determine the source of difference.

TABLE 2

DOD QSM VERSION 4.1 QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Method of standard additions (MSA)	When matrix interference is confirmed.	NA.	NA.	NA.	Document use of MSA in the case narrative.
Results reported between DL and LOQ	NA.	NA.	NA.	Apply J-flag to all results between DL and LOQ.	

TITLE: DIGESTION AND ANALYSIS OF AQUEOUS SAMPLES FOR MERCURY BY USEPA METHOD 7470

TABLE 3

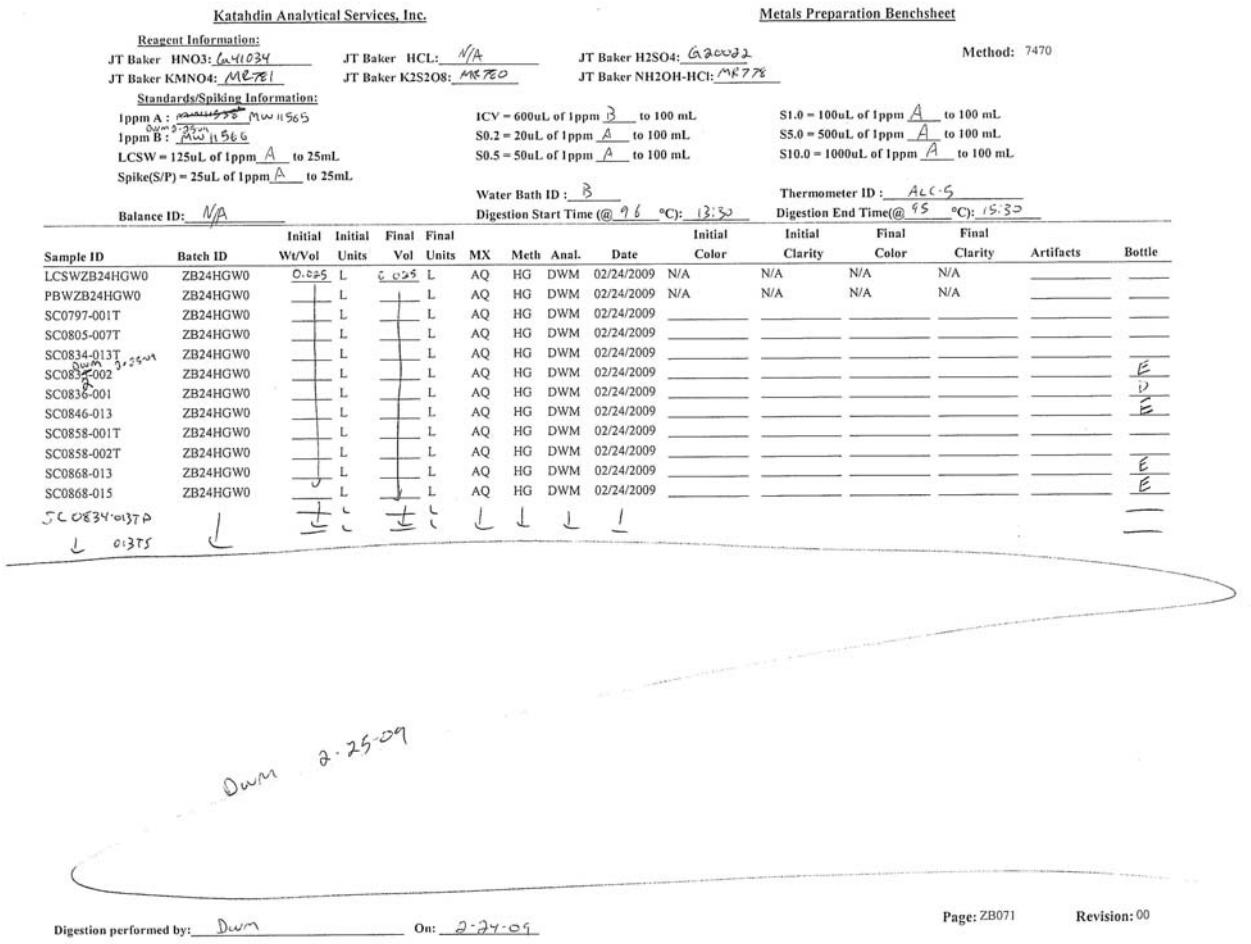
SUMMARY OF METHOD MODIFICATIONS

TOPIC	KATAHDIN SOP CA-615-04	USEPA METHOD 7470
Reagents	Stannous chloride dissolved in hydrochloric acid to prevent clogging of mercury analyzer, per instrument manufacturer's recommendation.	Stannous chloride dissolved/suspended in sulfuric acid.
Procedures	1)Sampling and gas stream switching performed automatically by mercury analyzer. 2)Working Mercury standard prepared monthly in 2% nitric; calibration standards prepared fresh daily.	1)Sampling and gas stream switching performed manually by analyst. 2)Working Mercury standard prepared fresh daily and acidity maintained at 0.15% nitric.
QC – Calibration Verification	1) Known reference sample (ICV) analyzed daily. 2) Calibration verified after every 10 samples with CCV.	1) Known reference sample analyzed quarterly. 2) Calibration verified after every 20 samples.
QC - Calibration Blanks	Acceptance criteria employed for 245.1: ± PQL	Acceptance criteria stated in 245.1: ± MDL

TITLE: DIGESTION AND ANALYSIS OF AQUEOUS SAMPLES FOR MERCURY BY USEPA METHOD 7470

FIGURE 1

EXAMPLE PAGE FROM MERCURY PREPARATION LOGBOOK



Katahdin Analytical Services, Inc. — Metals Preparation Benchsheet

Reagent Information:
JT Baker HNO3: La41034 — JT Baker HCL: N/A — JT Baker H2SO4: G20002 — Method: 7470
JT Baker KMNO4: MR781 — JT Baker K2S2O8: MR780 — JT Baker NH2OH-HCl: MR778

Standards/Spiking Information:
1ppm A : ~~MW11538~~ MW11565
1ppm B : Dwm 2.25.09 MW11566
LCSW = 125uL of 1ppm A to 25mL
Spike(S/P) = 25uL of 1ppm A to 25mL
ICV = 600uL of 1ppm B to 100 mL
S0.2 = 20uL of 1ppm A to 100 mL
S0.5 = 50uL of 1ppm A to 100 mL
S1.0 = 100uL of 1ppm A to 100 mL
S5.0 = 500uL of 1ppm A to 100 mL
S10.0 = 1000uL of 1ppm A to 100 mL
Balance ID: N/A
Water Bath ID : B
Digestion Start Time (@ 96 °C): 13:30
Thermometer ID : ALC-5
Digestion End Time(@ 95 °C): 15:30

Sample ID	Batch ID	Initial Wt/Vol	Initial Units	Final Vol	Final Units	MX	Meth	Anal.	Date	Initial Color	Initial Clarity	Final Color	Final Clarity	Artifacts	Bottle
LCSWZB24HGW0	ZB24HGW0	0.025	L	0.025	L	AQ	HG	DWM	02/24/2009	N/A	N/A	N/A	N/A		
PBWZB24HGW0	ZB24HGW0		L		L	AQ	HG	DWM	02/24/2009	N/A	N/A	N/A	N/A		
SC0797-001T	ZB24HGW0		L		L	AQ	HG	DWM	02/24/2009						
SC0805-007T	ZB24HGW0		L		L	AQ	HG	DWM	02/24/2009						
SC0834-013T	ZB24HGW0		L		L	AQ	HG	DWM	02/24/2009						
SC0834-002 (Dwm 2.25.09)	ZB24HGW0		L		L	AQ	HG	DWM	02/24/2009						E
SC0836-001	ZB24HGW0		L		L	AQ	HG	DWM	02/24/2009						E
SC0846-013	ZB24HGW0		L		L	AQ	HG	DWM	02/24/2009						E
SC0858-001T	ZB24HGW0		L		L	AQ	HG	DWM	02/24/2009						
SC0858-002T	ZB24HGW0		L		L	AQ	HG	DWM	02/24/2009						
SC0868-013	ZB24HGW0		L		L	AQ	HG	DWM	02/24/2009						E
SC0868-015	ZB24HGW0	J	L	J	L	AQ	HG	DWM	02/24/2009						E
SC0834-013TD	"	"	"	"	"	"	"	"	"						
" 0.375	"	"	"	"	"	"	"	"	"						

Dwm 2.25.09

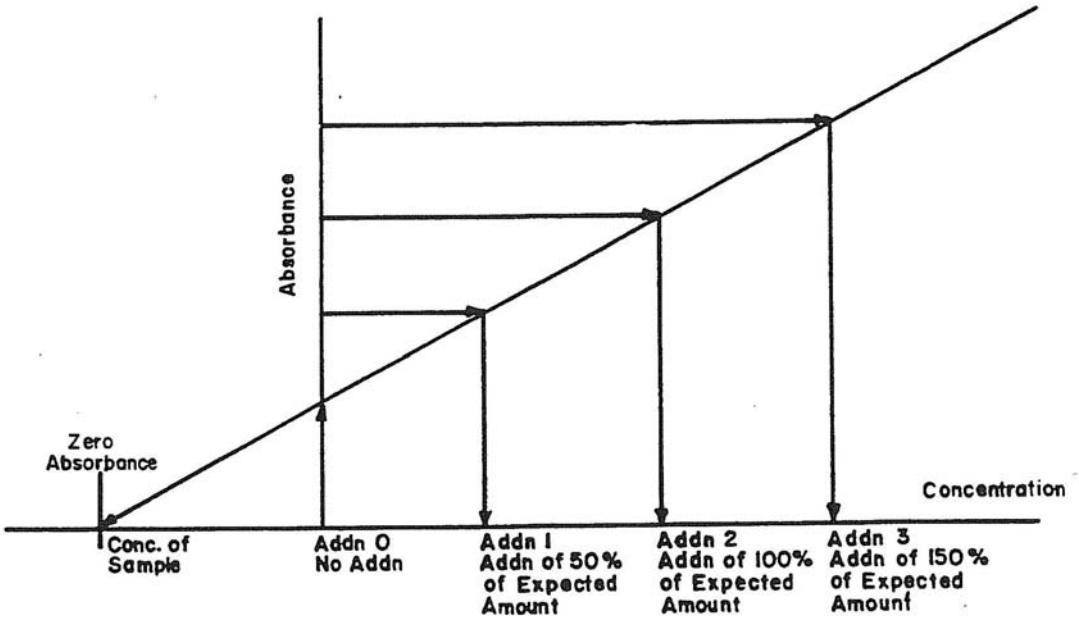
Digestion performed by: Dwm On: 2-24-09

Page: ZB071 Revision: 00

TITLE: DIGESTION AND ANALYSIS OF AQUEOUS SAMPLES FOR MERCURY BY USEPA METHOD 7470

FIGURE 2

STANDARD ADDITIONS PLOT



KATAHDIN ANALYTICAL SERVICES, INC.
STANDARD OPERATING PROCEDURE

SOP Number: SD-902
Revision History
Cover Page
Page 1

TITLE: **SAMPLE RECEIPT AND INTERNAL CONTROL**

Prepared By: Andrea Colby Date: 6/2002

Approved By:

Group Supervisor: Andrea Colby Date: 6/6/02

Lab Operations Mgr: J. C. Burton Date: 6/5/02

QA Officer: Deborah J. Nadeau Date: 6.6.02

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
04	Changed cover sheet, minor changes to sections 7.1, 7.6, 7.7.4, 7.10 + 7.20. Complete rewrite of sections 7.11 + 7.12 to comply with new KIMS	DN	6.6.02	6.6.02
05	Added verbal date entry to KIMS. Added reference to immediate internal COC book. Added Department Manager reference. Added section 7.7.3. Updated new incoming	DN	05.04	05.04
06	Added procedure + logbook page for checking turbidity of drinking water samples. Changed wet chem shorts board to a book (included example page). Added custody procedures for food/micro. Added VOA soil freezer storage.	DN	01.26.04	01.26.04
07	Added instructions to create lettered labels. Changed sample locations to reflect new building. Removed Figures B and 10. Updated Table and Figures w/ current ones. Added wording to sect. 7.7.5 to clarify how pH measurements are taken.	LAD	02/07	02/07
08	Added summary stating sample acceptance policy. Deleted all references to radiation checks (not performed). Add IR gun usage. Reorganized section 7.0 to prioritize time sensitive tasks. Added wireless thermometer monitoring. Updated SRCR. Other minor changes. Added section concerning locking of coolers. Added more detail to 7.18 on unique container IDs. Added more detail on immediate COCs & a section on retention of samples.	DN	~~05/09~~ 08/09 8.4.09	~~05/09~~ 08/09

TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

Please acknowledge receipt of this standard operating procedure by signing and dating both of the spaces provided. Return the bottom half of this sheet to the QA Department.

I acknowledge receipt of copy ___ of document **SD-902-08**, titled **Sample Receipt and Internal Control**.

Recipient: _____ Date: _____

KATAHDIN ANALYTICAL SERVICES, INC.
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy ___ of document **SD-902-08**, titled **Sample Receipt and Internal Control**.

Recipient: _____ Date: _____

TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

1.0 SCOPE AND APPLICATION

Katahdin Analytical Services, Inc. requires the use of specific receiving, acceptance, identification, storage, and distribution procedures for samples it accepts for analyses. These procedures assure that:

- samples are uniquely identified,
- samples are protected from loss or damage,
- essential sample characteristics are preserved,
- any alteration of samples (e.g., filtration, preservation) is documented,
- the correct samples are analyzed, and
- a record of continuous sample custody and utilization is established.

The purpose of this SOP is to describe the procedures used for the receipt and tracking of samples received by Katahdin Analytical Services, Inc. (Katahdin).

1.1 Definitions

SDG: Sample Delivery Group – A group of samples to be reported as one data package.

1.2 Responsibilities

It is the responsibility of all Katahdin staff who receive samples or handle samples in the course of analysis to follow the procedures set forth in this SOP, to document their understanding of the procedures in their training files (refer to Katahdin SOP QA-805, current revision, "Personnel Training & Documentation of Capability"), and to suggest changes and revisions when appropriate. All technical staff are responsible for monitoring their immediate areas, stopping an activity when a problem is detected or suspected, initiating corrective action when needed, documenting any actions taken, and notifying the appropriate individual (e.g., Department Manager, Operations Manager, QAO). The primary re sponsibility for implementing real-time corrective actions and maintaining an effective QA self-inspection system resides with Katahdin staff. When problems are identified Katahdin personnel are expected to attempt to resolve situations within the scope of their technical knowledge, and to seek assistance from peers and the Department Manager as necessary.

It is the responsibility of Department Managers to oversee the adherence to Katahdin QC practices and internal documentation of laboratory activities within their area, to take corrective actions where needed and communicate problems to the Operations Manager, QAO or Vice President/President when warranted.

It is the responsibility of the Operations Manager to oversee adherence to Katahdin QA/QC practices by all laboratory groups under his/her authority, to help identify

problems and assure resolution, to facilitate corrective action where needed, and to communicate problems and concerns to the QAO and Vice President/President.

It is the responsibility of the Quality Assurance Officer (QAO) to oversee adherence to this SOP, to conduct periodic audits of each laboratory, to track corrective action reports, resolution, and documentation, and to communicate concerns and report findings to the Vice President/President. The QA Officer shall function independently from laboratory operations and be able to evaluate data objectively and perform assessments without outside influence. The QA Officer has the authority to independently halt production operations (including data reporting) if warranted by quality problems.

1.3 Safety

Users of this procedure must be cognizant of inherent laboratory hazards, proper disposal procedures for contaminated materials and appropriate segregation of hazardous wastes. The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical should be treated as a potential health hazard. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Everyone involved with the procedure must be familiar with the MSDSs for all the materials used in this procedure.

Each qualified analyst or technician must be familiar with Katahdin Analytical safety procedures and the Katahdin Environmental Health & Safety Manual and must follow appropriate procedures. These include the use of appropriate personal protective equipment (PPE) such as safety glasses, gloves and lab coats when working with chemicals or near an instrument and not taking food or drink into the laboratory. Each analyst should know the location of all safety equipment. Each analyst shall receive a safety orientation from their Department Manager, or designee, appropriate for the job functions they will perform.

1.4 Pollution Prevention/Waste Disposal

Whenever possible, laboratory personnel should use pollution prevention techniques to address their waste generation. Refer to the current revision of the Katahdin Hazardous Waste Management Program for further details on pollution prevention techniques.

Wastes generated during the receipt of samples must be disposed of in accordance with the Katahdin Environmental Health & Safety Manual and SOPs SD-903, “Sample Disposal” and CA-107, “The Management of Hazardous Waste as it Relates to the Disposal of Laboratory Process Waste, Reagents, Solvents and

Standards,” current revisions. Expired standards are placed in the Katahdin hazardous waste storage area, and disposed of in accordance with these SOPs.

TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

2.0 SUMMARY OF METHOD

Regulatory, program, and/or method requirements dictate the specifics of sample acceptance. These requirements include, but are not limited to, temperature upon receipt, chemical preservation, container type, sample amount, holding time considerations and complete and accurate documentation of all of these conditions, as well as sample identification. Katahdin's sample acceptance policy is to note any anomalies, discrepancies or non-compliances concerning the receipt of samples. The client is always notified with these issues to direct Katahdin on how and whether to proceed with analysis. All guidance from the client is recorded in the project phone logs and/or on the Sample Receipt Condition Report, which becomes part of the final report. Conditions or analyses performed which do not meet the necessary requirements are narrated or notated as described in the individual analytical SOPs.

3.0 INTERFERENCES

Not applicable.

4.0 APPARATUS AND MATERIALS

4.1 Thermometer – Oakton® Non-Contact Infrared Thermometer, or equivalent, capable of reading 0.1°C and digital probe style capable of reading 0.1°C (used for back-up).

4.2 Capillary tubes – 75 mm Hematocrit Tubes, disposable

4.3 Wide range pH test strips, pH 0 to 14 pH, EMD ColorpHast or equivalent.

4.4 Narrow range pH test strips, pH 0 to 2.5 pH, EMD ColorpHast or equivalent.

4.5 Narrow range pH test strips, pH 11 to 13 pH, EMD ColorpHast or equivalent.

5.0 REAGENTS

Preservatives - refer to Table 1, Sampling and Preservation Requirements, for specifics.

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

Refer to Table 1, Sampling and Preservation Requirements, for specifics.

7.0 PROCEDURES

PROCEDURES FOR SAMPLE CUSTODIAN

The following procedures include all steps to be completed for satisfactory receipt and acceptance of samples at Katahdin. These steps do not necessarily have to be performed in the exact order as described. Sample deliver ies occur constantly throughout the day, so the sample custodian must multi-task and move back and forth between different procedures to accomplish the most critical tasks of checking receipt temperatures and checking for "RUSH" or quick hold time parameters.

7.1 When samples (except for non-environmental food samples) are dropped off, by either a delivery service (i.e. FEDEX or UPS) or by the client, the Chain-of-Custody (COC) should be signed immediately. The client (who is delivering or that has shipped samples with a delivery service) shall sign (at the lab upon delivery or prior to shipment of samples) that they have relinquished custody to the laboratory. The laboratory shall sign and record the date and time that custody is accepted. (Refer to Figures 1-3 for a Katahdin standard COC, a Katahdin Homeowner COC, and a Katahdin Food/Microbiology COC).

7.2 Cut custody seals and open all coolers. Remove the packets containing the client Chains-of-Custody (COCs).

7.3 Using the COCs, enter the date and time of sample receipt and the client name into the next available work order/login number in the sample receipt logbook (Figure 4). Initial each entry (line) to maintain a record of the individual who assigned each group of samples a discreet lab work order/login number. Record the assigned work order numbers in the appropriate space on the client COCs. Complete the log-in entry date and time once samples are logged in as described below.

7.4 Inventory the COCs for any "RUSH" or quick hold time analyses. Notify the appropriate section managers of these analyses. List any samples for analyses that have short hold times in the "Wet Chemistry Shorts and Rushes Logbook" (Figure 5) in the wet chemistry laboratory. Be sure to list the client, number of samples and date and time of the earliest sample. GC or GC/MS personnel must be informed when ENCORES are received so that they may be scheduled for extrusion. Microbiology personnel should also be informed of any microbiology samples that arrive. Parameters that routinely require short analytical hold times are:

Coliforms	Color	pH
Nitrate/Nitrite	Dissolved Oxygen	Turbidity
Ferrous iron	Orthophosphate	Hex. Chromium
MBAS	TBOD	Free CO_2
Sulfite	ENCORE soil samples	Settleable Solids
Odor	Residual Chlorine	CBOD

7.5 Inspect the condition of custody seals, cooler, ice condition and samples received. Note any non-intact conditions on the Sample Receipt Condition Report (SRCR - Figure 6). Notify the Katahdin project manager (PM) of any discrepancies or problems with sample receipt. The PM contacts the client as necessary. If breakage of a potentially hazardous sample is discovered, close and seal the packing container with all the samples inside and move to a hood in the organic extractions area or to the smaller hood in the login area if space permits. One of the three Katahdin Emergency Response Coordinators or the Katahdin Environmental Health & Safety Manager must be notified. Disposition of the broken and other possibly contaminated samples will be determined on a case-by-case basis in accordance with the laboratory's handling procedures for hazardous waste as outlined in the Katahdin Environmental Health & Safety Manual. Generally, when a sample has broken and has mixed with any ice in the cooler, that liquid will be poured off into 2 liter plastic containers and labeled as "do not use". These containers will be disposed of as soon as the disposition of the appropriate samples has been determined through analysis.

7.6 If there is no breakage of a potentially hazardous sample:

Check cooler temperatures using the IR thermometer assigned to the sample receipt area. If a cooler temperature blank is present, aim the IR gun at the temperature blank; otherwise aim the IR gun at any sample in the cooler if no temperature blank is present. Be sure that the IR gun is within 6 inches of the bottle and not aimed at a label on the bottle. Press the trigger on the handle and be sure the red dot is visible on the bottle surface. The IR gun has been set to read in degrees celcius. If checking the temperature of a plastic bottle, set the emissivity at 0.90. If checking the temperature of a glass bottle (either amber or clear), set the emissivity at 0.85. Refer to Figure 7 for manufacturer's instructions on changing the emissivity. Record the temperature on the Sample Receipt Condition Report. Receipt temperatures should be <6 °C, without freezing. Any temperature falling outside of this range must be noted on the SRCR and reported to the appropriate Katahdin project manager.

Note: Samples received for metals analysis only do not have to meet any temperature receipt requirements.

Note: A probe type thermometer is retained as back-up in case there is a problem with the IR thermometer.

7.7 Note the condition of the ice or ice packs. If the ice has melted and the temperature is out of acceptance criteria, note this on the SRCR. For samples that are hand delivered to the laboratory immediately after collection (i.e. sample collection times are <6 hours old), the temperature blank and/or cooler temperature will most likely not meet the acceptance criteria. The samples shall be considered acceptable if there is evidence that the chilling process has begun such as arrival on ice. Note this on the SRCR. If samples (that were just collected) have not arrived on ice, note this on the SRCR, and start the cooling process as soon as possible after arrival at the laboratory.

Note: All clients must be notified when samples are received that do not meet the appropriate temperature requirements. In these cases, certain regulatory requirements may not be met and may invalidate certain data.

7.8 Inventory the samples against the chain of custody (COC). If the COC is incomplete, the sample custodian must inform the appropriate Katahdin project manager (PM). The PM may make changes to correct or complete the COC, but all changes must be initialed and dated. Changes must be noted on the SRCR. Any discrepancies between the samples and the COC must also be noted on the SRCR.

7.9 Using the Sampling and Preservation Requirements Table (Table 1) as a reference, check if samples are in proper containers and received correct pretreatment (e.g., filtration, preservation) for the analyses requested. For aqueous parameters requiring preservation, check pH by inserting a clean capillary tube into the sample and dabbing the tube on wide range pH paper. If the pH is not clearly either less than 2 or greater than 12, the appropriate narrow range pH paper must be used. NOTE: The pH of volatile organic (VOA) samples is checked and recorded by the analyst after completion of analysis and not by sample receipt personnel. The used capillary tube is discarded and a new capillary tube is used for each sample.

Additional preservative is added to samples if the pH is not in the range specified in the Sampling and Preservation Requirements Table. No more than 10% of the original sample volume should be added as preservative. If the client has noted that the sample reacts violently (i.e., foams and bubbles) upon preservation, add no more preservative to the sample. Some clients may wish to be contacted if their samples are found to be improperly preserved. Record all preservation discrepancies on the Sample Receipt Condition Report including the lot number of the preservative added. If additional preservative is added, a sticker with the type of preservative must be placed on the sample container.

Note: Preservatives are obtained from the larger containers in the bottle preparation area.

Note: If samples are received unpreserved for 200.7 or 200.8 analysis, the samples must be preserved to pH <2 with nitric acid. Samples must be held for 16 hours after preservation before sample preparation can begin.

7.10 For samples requiring filtration as pretreatment (i.e. for dissolved metals), the work order/login numbers are recorded in the filtration logbook (see Figure 8). The samples are filtered by the Metals Group.

7.10.1 A 500 mL filter flask and filter funnel are acid rinsed three times in a 10% nitric acid bath, then three times with Laboratory Reagent Grade Water.

7.10.2 A vacuum pump is attached.

7.10.3 A 0.45 micron filter is rinsed three times with 5% nitric acid and three times with Laboratory Reagent Grade Water. The rinsate is discarded.

7.10.4 A sufficient sample aliquot is filtered and preserved with concentrated nitric acid to pH <2.

7.10.5 The bottles are labeled with the work order/login number and other sample information and stored at <6 ° C until the time of digestion.

7.11 Using the Sampling and Preservation Requirements Table (Table 1) as a reference, determine if sufficient volume of sample is present for analysis. Note discrepancies on the SRCR.

7.12 For drinking water samples, enter the appropriate information (work order, date, etc.) into the Measured Turbidity and Preservation of Incoming Samples Logbook. Inform the appropriate analyst of the sample. The turbidity must be measured prior to sample preparation. If the turbidity is <1 NTU, the sample does not have to be digested prior to metals analysis. If the turbidity is >1 NTU, the sample must be digested prior to metals analysis. The sample must be preserved after the turbidity measurement is taken. Record the appropriate information in the logbook (Figure 9).

7.13 Notify the PM immediately if there are any discrepancies or problems with sample receipt. The PM will contact the client for information and resolution as necessary. All decisions to proceed or not to proceed with analysis associated with samples received that do not meet specified acceptance criteria (i.e. cooler temperature, preservation, container, etc.) must be fully documented on the SRCR. Although this form is included with all client reports, additional narration or flagging of data may be necessary.

7.14 Review any additional paperwork that accompanies the sample(s) submitted for analysis along with laboratory-generated information. This includes shipping forms, letters, chain-of-custody forms, sample labels, Incoming Sample Information Sheets (ISIS), quotes, memos, etc. These forms may provide details on specific client requests. The ISIS will provide information on specifics for log-in. Refer to Figure 10 for an example.

7.15 Resolve any questions or concerns raised by steps 7.1-7.14 by consulting the correspondence files or client services personnel or communicating directly with the client. Note in the notes section of the SRCR any deviations from normal sample handling or analytical procedures (e.g., client requests analysis although hold-time expired).

7.16 When non-environmental food samples are delivered to the laboratory, they are taken immediately to the food/microbiology laboratory and stored in the refrigerators there. A copy of the Chain-of-Custody is left with the analysts. The original paperwork is forwarded to sample log in where the job is logged into the KIMS system.

7.17 The following information is documented via the Katahdin Information Management System (KIMS) and a work order/login COC report (Figure 11) is generated for the samples received:

7.17.1 Log onto KIMS by entering employee ID under "Username", employee specific password under "Password" and KIMS under "Database".

7.17.2 Once logged onto KIMS select "Sample Management" and then "Login".

7.17.3 Select "New" and the next available Login ID number will automatically be entered. Select "OK" and the Sample Definition screen will open.

Note: If a Work Order number has already been opened, select "change" and type in the appropriate number to access the information.

7.17.4 In the Sample Definition Screen, enter the following information.

Client ID -	Enter client sample description.
ReceiveDate -	Enter in date that samples were received in the lab in the format YY-Month-DD.
CollectDate -	Enter in date that samples were collected in the format YY-Month-DDTIME.
TAT -	Enter TAT for hardcopy report.
DueDate -	Due date will automatically be calculated based on calendar days.
VerbalDate -	Manually type in verbal due date.
QuoteRef -	Enter quote number if applicable.
Project -	Enter project number if applicable.
Account -	Enter client specific account number.
Account Name -	Account name will automatically be entered.
Collected By -	Enter name/initials of sampler listed on COC. If unknown, enter "Client".
Locator -	May be used for client ID information when requested by the project manager.
Site -	Enter project site name.

Description -	May be used for long client Ids when requested by the project manager.
Discount -	No entry-not currently used.
Priority -	No entry-not currently used.
Fact. -	No entry-not currently used.
Expected -	No entry-not currently used.
Comments -	Enter MS/MSD, verbal due date and any sample irregularities if applicable.
OrderDate -	Current date is automatically entered.
Matrix -	Enter sample matrix code where

AQ = Aqueous
SL = Solid, Soil, Sludge
FP = Free Product
WP = Wipe
NOAQ = NonAqueous
DW = Drinking Water
SLD = Food Solid
AR = Air
SWAB = Swab
SAL = Saline
TIS = Tissue

Product Code -	Enter analysis code per test requested on COC.
Type -	Product code type will automatically be entered where

S = Stand alone
P = Parent
C = Children

Fact. -	No entry-default is 1.
Price -	This is left as is by sample log-in. During project management review of the work order, the prices are entered based on quotes or standard prices.
Cost -	No entry needed.
Lev -	No entry needed.
Type -	Container type will automatically be entered.
Bot -	Enter number of containers per test for printing of labels.

Login Info - Parameter Data Screen will open. Enter following information

KAS Proj. Manager- Initials of Katahdin person overseeing the project.
Client PO#- Client purchase order.
Project- Project name.
Cooler Temperature- Temperature blanks or cooler temps.
Delivery Services- Method of delivery to the lab.
QC Level- QC Level of report and regulatory agency (ie., IV NFESC).
SDG ID- Sample Delivery Group ID if applicable.
SDG Status- Begin, Continue or End.
Analysis Instructions- PM will enter special instructions regarding project.
Report Instructions- PM will enter special instructions regarding project.
Regulatory List- Used for federal programs.
EDD Format- Specific KAS EDD format.

Select “SAVE” and then “CANCEL”.

Addresses - Select “Addresses” and the Address Links screen will open. The billing address is the default address of the account. Enter the client account code under “Project/Account” and select the report to contact under “Address Type”. Select the appropriate boxes for report, report CC and invoice CC. Select “SAVE” and then “CLOSE”.

Create Containers - Select “Create Containers”. Letters will be assigned to each sample number. Select “OK” until letters have been assigned to each sample number. To manually assign letters, Select “Enter Container IDs” and “OK”. Enter sample numbers including letters and select “OK”, “Close”, “Yes” to save changes, “Cancel” and “Cancel”.

7.17.5 To print the login report, select “Reports”, “Login” and “Login COC”. Enter login number under “Login Number”. Select “OK”, “Run Report” and then “Print”.

7.18 To print labels unique to each bottle, select “Reports”, “Login” and “Labels”. Enter login number under “Login/Prelogin”, select “Background (IDX L)” and select F9 on keyboard under “Select Sample Label”. Select “OK” and then “Print”. After labels print out select “Cancel”.

Note: As stated in “create containers” above, each sample bottle is assigned a unique ID. The job is given a work order number. Each different client sample ID is given a numerical number following the work order number and each sample container with the same client ID is given a container ID using alphabetical letters. This series of work order, sample number and container ID is transcribed throughout the raw data for traceability purposes.

Example: One job containing one client sample with 3 different containers:

SC9001-001(a), SC9001-001(b), SC9001-001(c)

Example: One job containing two client samples with 2 different containers for each:

SC9002-001(a), SC9002-001(b), SC9002-002(a), SC9002-002(b)

7.19 Affix permanent sample number labels to sample containers, assuring that sample IDs on labels correspond to sample bottle IDs. Do not obscure client ID on the bottles.

7.20 Place samples in their designated storage locations and log them in, noting initials, date and time, work order/login and sample numbers, and storage location on the internal laboratory chain of custody form (Figure 12). Place form in the appropriate binder in the log in area. Non-environmental food samples do not get an internal COC and are taken immediately to the food/microbiology lab for storage.

Storage location of the samples is determined by type of sample and/or type of analysis, as outlined below. Most samples are stored in the walk-in cooler, which is organized by test type and work order/login number.

Specific storage locations are described below.

7.20.1 Aqueous samples for wet chemistry (except hardness, see 7.19.4 below) - left aisle, both sides, as you enter walk-in cooler. TOC vials are to be stored in the trays designated for TOC samples.

7.20.2 Aqueous samples for organic extractions – right aisle, left side, as you enter walk-in cooler.

7.20.3 Non-aqueous samples (all analyses except volatile organics) - to the right and towards the back as you enter walk-in cooler. Non-aqueous samples for

volatile organics are stored in "VOA Refrigerator 2" located in the Volatiles Laboratory.

7.20.4 Aqueous samples for metals and/or hardness analyses – right aisle, right side towards the front as you enter walk-in cooler.

7.20.5 Samples (aqueous and solid) for volatile organics analyses (VOA) – All aqueous samples and soil samples in VOA vials (preserved with methanol or sodium bisulfate) are stored in "VOA Refrigerator 1" in the Volatiles Laboratory. VOA soils in jars or ENCORE samplers are stored in "VOA Refrigerator 2" in the Volatiles Laboratory. VOA samples known or suspected to be hazardous (such that cross-contamination of other samples might occur) are placed in a "paint can" and stored in the walk-in.

7.20.6 Soil samples for volatile organics analyses (VOA) that are unpreserved or preserved with Laboratory Reagent Grade Water are stored in "VOA Freezer 1" in the volatiles laboratory.

Sample storage coolers are not locked, but internal chain-of-custody is documented with respect to native samples, extracts and digestates within the laboratory. The laboratory maintains a secure facility with respect to unauthorized personnel, as described in the current revision of Katahdin SOP, AD-004, Laboratory Facility Security and Confidentiality. All sample storage coolers are equipped with locks if specific project or regulatory requirements deem it necessary.

7.21 Sample Receipt gives the Work order/login COC report and confirmation of the job, as logged-in, to the appropriate Katahdin project manager. All chain-of-custody and other receipt documentation must accompany the job. The project manager reviews the job for accuracy and completeness. Any unresolved issues should be resolved at this time. Any project or program specific forms should be included with the paperwork at this time. These forms may include CLP forms or state-specific forms. The project manager then dispatches the work order/login to the individual department worklists. The dispatched work order/login package is then filed in Data Management where the complete package will eventually be compiled.

7.22 The temperature of all sample storage refrigerators and freezers is recorded daily by assigned individuals. Notebooks containing a record of each refrigerator and freezer temperature history are used for this purpose and are maintained by the assigned individuals. Temperatures above or below the acceptance range are to be brought to the attention of a Department Manager, O perations Manager, or Quality Assurance Officer. Such an occurrence and the actions taken to correct it must be noted in the comments column of the temperature recording notebook next to the temperature measurement. (See Figure 13).

Additionally, temperatures of storage units are monitored continuously by wireless thermometers. A temperature is recorded electronically every 10 minutes. The QAO

can generate a specified report as needed, including every reading or maximum/minimum temperatures for a given timeframe. These monitoring devices ensure continual compliance seven days per week. The data can be used to check for problems.

PROCEDURES FOR CHEMISTS

7.23 When removing a sample from its storage location, record on the laboratory internal chain-of-custody (from the appropriate department) the sample number, date and time it was removed, chemist who removed it, and the analysis to be conducted or reason for removal.

7.24 If the samples have not been logged in yet and they need to be pulled in order to analyze short holding time parameters, the analyst taking the sample must use the designated logbook (Immediate Internal COC – Figure 14) to sign the samples out. Many circumstances lead to analysts having to pull samples before they are logged into the KIMS system. It is everyone's responsibility to ensure that all samples can be accounted for at all times. Failure to do so can create confusion and bottle necks for others trying to access the samples. Samples that are pulled before log-in must be returned to the designated bin in the sample receipt area. When the logbook for Immediate Internal COC's is used, the standard internal COC's do not have to be signed at a later date. The Immediate Internal COC Logbook must always be consulted if there is ever a question about whether an internal COC has been completed.

7.25 If a sample is not consumed by an analysis, return the remaining sample to its assigned storage location and enter the date and time returned on the laboratory internal chain-of-custody record.

7.26 If analysis consumes the entire sample, indicate this on the laboratory internal chain-of-custody record.

7.27 After the completion of all analyses, the original "left over" sample containers will remain in sample storage until their final disposal. Samples are held during this period for the purposes of retesting if required by a laboratory corrective action or by a client. Refer to the current revision of Katahdin SOP, SD-903, Sample Disposal, for details on final disposal of samples.

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

Each thermometer used to monitor sample storage or cooler temperatures must be calibrated annually against a NIST traceable thermometer. The QAO is responsible for ensuring that the thermometer(s) are scheduled for annual calibration and for maintaining the calibration records. All other procedures and documentation listed in this SOP must be followed at all times.

9.0 METHOD PERFORMANCE

Not applicable.

10.0 APPLICABLE DOCUMENTS/REFERENCES

"Handbook for Analytical Quality Control in Water and Wastewater Laboratories," U.S. EPA EMSL Office of Research and Development, March 1979.

Code of Federal Regulations 40, Parts 136 and 141.

"Test Methods for Evaluating Solid Waste: Physical/Chemical Methods," SW-846 Chapters 1 & 2, USEPA, Third Edition, including Updates I, II, IIA, and IIB, III June, 1997.

Katahdin Analytical Services, Inc., Environmental Health & Safety Manual, current revision.

LIST OF TABLES & FIGURES

TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

TABLE 1

SAMPLING AND PRESERVATION REQUIREMENTS

PARAMETER – AQUEOUS MATRICES	METHOD	QUANTITY	CONTAINER	PRSV	HOLD TIME
GENERAL CHEMICAL ANALYSES					
Acidity	305.1	100 mL	P,G	1,2	14 days
Alkalinity-Manual Titrimetric	310.1	100 mL	P,G	1,2	14 days
Ammonia-Nitrogen with distill-Auto. Phenate	350.1	1 L	P,G	1,3	28 days
Ammonia-Nitrogen-Automated Phenate	350.1, 350.2	250 mL	P,G	1,3	28 days
Anions (Cl, Br, SO4, NO2, NO3)	300.0	250 mL	P, G	1	48hr/28days
Bicarbonate, Carbonate (see pH & alkalinity)	calc.				
Biochemical Oxygen Demand-Carbonaceous	405.1	1 L	P,G	1	48 hours
Biochemical Oxygen Demand-Total	405.1	1 L	P,G	1	48 hours
Bromide	320.1	500 mL	P,G	1	28 days
Chemical Oxygen Demand-Manual Colorimetric	410.4	100 mL	P,G	1,3	28 days
Chloride-Automated Ferricyanide	325.2	100 mL	P,G	1	28 days
Chlorine, Residual	SM4500-Cl G	100 mL	P,G	1,9	ASAP
Chromium, Hexavalent	SM3500Cr D / SW7196	200 mL	P,G	1,9	24 hours
Color, Apparent	110.2	100 mL	P,G	1,2	48 hours
Cyanide, Amenable-Spectrophotometric	335.1	250 mL	P,G	1,5	14 days
Cyanide, Total-Spectrophotometric	SM4500CN C 335.3, 335.4	250 mL	P,G	1,5	14 days
Dissolved Oxygen(Lab)-Membrane Electrode	360.1	500 mL	G	1	ASAP
Ferrous Iron - Colorimetric	SM3500-Fe D	250mL	P	1	24 hrs
Fluoride with distillation, Potentiometric ISE	SM4500F C/340.2	500 mL	P only	1	28 days
Fluoride, Potentiometric ISE	340.2	200 mL	P only	1	28 days
Free CO_2	SM4500-CO_2 C	250mL	P	1	24 hrs.
Hardness, Total-Manual Titrimetric	130.2,SM2340C	250 mL	P,G	4	6 months
MBAS, Extraction-Colorimetric	SM5540C	1 L	P,G	1	48 hours
Nitrate+Nitrite-Automated Cadmium Reduction	353.2	100 mL	P,G	1,3	28 days
Nitrate-Automated Cadmium Red./Diazotization	353.2	100 mL	P,G	1	48 hours
Nitrite-Automated Diazotization	353.2	100 mL	P,G	1	48 hours
Oil & Grease-Total Recoverable, Gravimetric N-Hexane extractable material N-Hexane extractable material w/ silica gel cleanup	1664	(2) 1 L	glass only	1,11	28 days
pH (Laboratory)	150.1	100 mL	P,G	1,2	24 hours
Phenolics, Total Recoverable-Manual 4AAP	420.1	1000 mL	glass only	1,3	28 days
Phosphate, Ortho- Ascorbic Acid	365.2	100 mL	P,G	1	48 hours
Phosphate,Total	365.4	100 mL	P,G	1,3	28 days
Solids-Filterable Residue (TDS),Gravimetric180	160.1	250 mL	P,G	1	7 days
Solids-Nonfilterable Residue (TSS)	160.2	500 mL	P,G	1	7 days
Solids-Settleable Solids (SS)	160.5	1 L	P,G	1	48 hours

TABLE 1 (cont.)

SAMPLING AND PRESERVATION REQUIREMENTS

PARAMETER – AQUEOUS MATRICES	METHOD	QUANTITY	CONTAINER	PRSV	HOLD TIME
GENERAL CHEMICAL ANALYSES					
Solids-Total Solids	160.3	250 mL	P,G	1	7 days
Solids-Total Volatile (TVS)	160.4	250mL	P,G	1	7 days
Solids-Volatile Filterable Residue (VDS)	160.1/160.4	250 mL	P,G	1	7 days
Solids-Volatile Nonfilterable Residue (VSS)	SM 2540 F	500 mL	P,G	1	7 days
Specific Conductance-Wheatstone Bridge	120.1	100 mL	P,G	1,2	28 days
Sulfate-Turbidimetric	375.4	100 mL	P,G	1	28 days
Sulfide-Iodometric	376.1	500 mL	P,G	1,7	7 days
Sulfite-Titrimetric	377.1	500 mL	P,G	1,9	ASAP
Tannin/Lignin-Colorimetric	SM 5550 B	100 mL	P,G	1	7 days
TKN-Auto Block Digest, Spect.	351.2	100 mL	P,G	1,3	28 days
Total Inorganic Carbon	415.1	(2) 40 mL	VOA vial	1	28 days
Total Inorganic Carbon if with TOC	415.1	(2) 40 mL	VOA vial	1	28 days
Total Organic Carbon-Oxidation	415.1	(2) 40 mL	VOA vial	1,3	28 days
Total Organic Halogen	9020	500 mL	Amber Glass	1,3	28 days
Turbidity	180.1	100 mL	P,G	1	48 hours
ELEMENTAL ANALYSES					
Chromium, Hexavalent	7196/6010	500 mL	P,G	1,9	24 hrs
GFAA(Furnace) Elements	SM 3113/ 200 series	500 mL	P,G	4	6 months
ICP Elements	200.7/6010	500 mL	P,G	4	6 months
ICP MS Elements	200.8/6020	500 mL	P,G	4	6 months
Low Level Mercury	1631	500 mL	G	NA	90 days
Mercury	245.1/7470	500 mL	P,G	4	28 days
GC ORGANIC ANALYSES					
BTEX & MTBE	602 & 8021	(2) 40 mL	VOA vial	1,8,9	14 days(~)
EDB, DBCP & 1,2,3-TCP	504.1	(2) 40 mL	VOA vial	1,8,9	14 days(~)
Extractable Petroleum Hydrocarbons	MADEP/EPH	(2) 1000 mL	Amber Glass	12	14days/40days
Formaldehyde	556	(2) 40 mL	VOA vial	1,8,9	14 days(~)
Fuel Oil in Water	8015Modified	(2) 1000 mL	Amber Glass	1,8	7days/40days
Fuel Oil in Water	ME HETL 4.1.25	(2) 1000 mL	Amber Glass	1,8	7days/40days
Gasoline in Water	8015Modified	(2) 40 mL	VOA vial	1,8	14 days
Gasoline in Water	ME HETL 4.2.17	(2) 40 mL	VOA vial	1,8	14 days
Glycols	8015Modified	(2) 40 mL	VOA vial	1,8,9	14 days(~)
Herbicides	8151	(2) 1000 mL	Amber Glass	1	7days/ 40days
Methane, Ethane & ethene	RSK 175	(2) 40 mL	VOA vial	1,8,9	14 days(~)
PCB's (& Congeners)	608 & 8082	(2) 1000 mL	Amber Glass	1	7days/40days

TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

TABLE 1 (cont.)

SAMPLING AND PRESERVATION REQUIREMENTS

PARAMETER – AQUEOUS MATRICES	METHOD	QUANTITY	CONTAINER	PRSV	HOLD TIME
GC ORGANIC ANALYSES					
Pesticides	608 & 8081	(2) 1000 mL	Amber Glass	1	7days/40days
Pesticides and PCB's	608 & 8081/8082	(2) 1000 mL	Amber Glass	1	7days/40days
Purgeable Aromatics	602 & 8021	(2) 40 mL	VOA vial	1,8,9	14 days(~)
Purgeable Halocarbons	601 & 8021	(2) 40 mL	VOA vial	1,8,9	14 days(~)
Purgeables, Total	601 & 602	(2) 40 mL	VOA vial	1,8,9	14 days(~)
Purgeables, Total	8021	(2) 40 mL	VOA vial	1,8,9	14 days(~)
Solvents (Direct Injection)	8015M	(2) 40 mL	VOA vial	1	14 days
Volatile Petroleum Hydrocarbons	MADEP/VPH	(2) 40 mL	VOA vial	11	14days
GC/MS ORGANIC ANALYSES					
Acid Extractables-Priority Pollutants	625	(2) 1000 mL	Amber Glass	1	7days/40days
Acid Extractables-TCL	8270	(2) 1000 mL	Amber Glass	1	7days/40days
Base Neutral Extract.-Priority Pollutants	625	(2) 1000 mL	Amber Glass	1	7days/40days
Base Neutral Extractables-TCL	8270	(2) 1000 mL	Amber Glass	1	7days/40days
Drinking Water Volatiles - Low Level	524.2	(3) 40 mL	VOA vial	1,8,9,10	14 days(~)
PCB Homologues	680	(2) 1000 mL	Amber Glass	1	7days/40days
Polyaromatic Hydrocarbons	8270/8270 SIM	(2) 1000 mL	Amber Glass	1	7days/40days
Semivolatile Extractables-Priority Pollutants	625	(2) 1000 mL	Amber Glass	1	7days/40days
Semivolatile Extractables-TCL	8270	(2) 1000 mL	Amber Glass	1	7days/40days
Volatile Organics	8260	(2) 40 mL	VOA vial	1,8,9	14 days(~)
Volatile Organics-Priority Pollutants	624	(2) 40 mL	VOA vial	1,8,9	14 days(~)
HPLC ANALYSES					
HPLC-Explosives	8330, 8332	(2) 1000 mL	Amber Glass	1	7days/40days
MICROBIOLOGICAL ANALYSES					
Coliform, Fecal	SM 9222D, SM 9213D Mod.	100 mL	P,G	1,6	6 hours
Coliform, Total	SM 9222B	100 mL	P,G	1,6	30 hours
Coliform and E-coli, Total	SM9223B/Colitag	100 mL	P,G	1,6	30 hours
E-coli	SM9213D, Colilert/Quantitray	100 mL	P,G	1,6	6 hours
Heterotrophic Plate Count	SM9215B SIMPLATE	100 mL	P,G	1,6	30 hours

TABLE 1 (cont.)

SAMPLING AND PRESERVATION REQUIREMENTS

PARAMETER – SOLID MATRICES	METHOD	QUANTITY	CONTAINER	PRSV	HOLD TIME
GENERAL CHEMICAL ANALYSES		**4 oz=100 g**			
% Carbon	9060 mod.	4 oz	Soil Jar	1	28 days
Ammonia-Nitrogen-Automated Phenate	350.1 mod.	4 oz	Soil Jar	1	28 days (^)
Anions	9056	4 oz	Soil Jar	1	48hrs to 28 days from slurry (^)
Cation Exchange Capacity	9081	4 oz	Soil Jar	1	14days/7days (^)
Chloride-Automated Ferricyanide	9251/300.0	4 oz	Soil Jar	1	28days from slurry (^)
Cyanide, Amenable-Spectrophotometric	9012	4 oz	Soil Jar	1	14 days
Cyanide, Total-Spectrophotometric	9012	4 oz	Soil Jar	1	14 days
Fluoride, Potentiometric ISE	300.0 mod./340.2	4 oz	Soil Jar	1	28 days (^)
Lime Equivalency	310.1 mod.	4 oz	Soil Jar	1	28 days (^)
Nitrate+Nitrite-Automated Cadmium Reduction	300.0 mod./353.2	4 oz	Soil Jar	1	28 days (^)
Nitrate-Automated Cadmium Red./Diazotization	300.0 mod./353.2	4 oz	Soil Jar	1	48 hrs from slurry (^)
Nitrite-Automated Diazotization	300.0 mod./353.2	4 oz	Soil Jar	1	48 hrs from slurry (^)
Oil & Grease-Total Recoverable, Gravimetric N-Hexane extractable material N-Hexane extractable material w/ silica gel cleanup	9071	4 oz	Soil Jar	1	28 days (^)
Organic Nitrogen-Auto. Block Digest.,Spectro.	350.1/351.2 mod.	4 oz	Soil Jar	1	28 days (^)
pH (Laboratory)	9045	4 oz	Soil Jar	1	24 hours (^)
Phenolics, Total Recoverable-Manual 4AAP	Mod. 9065	4 oz	Soil Jar	1	28 days (^)
Phosphate, Ortho- Ascorbic Acid	300.0 mod./365.2	4 oz	Soil Jar	1	48 hrs from slurry (^)
Phosphate,Tot.-Auto Ascorbic Acid/Block Dig.	Mod. 365.4	4 oz	Soil Jar	1	28 days (^)
Solids-Ash	SM 2540 F	4 oz	Soil Jar	1	28 days (^)
Solids-Total Solids	CLP-CIP	4 oz	Soil Jar	1	28 days (^)
Solids-Volatile Solids	SM 2540 F	4 oz	Soil Jar	1	28 days (^)
Specific Conductance-Wheatstone Bridge	Mod. 9050	4 oz	Soil Jar	1	28 days (^)
Sulfate-Turbidimetric	9036/9038	4 oz	Soil Jar	1	28 days from slurry (^)
Sulfide-Iodometric	9030	4 oz	Soil Jar	1	7days from slurry (^)
Sulfide-Monier-Williams	40CFR-425	4 oz	Soil Jar	1	28 days (^)
Sulfite-Titrimetric	ASTM D3987/377.1 mod.	4 oz	Soil Jar	1	24 hrs from slurry (^)
TKN-Auto Block Digest,Spectro.	351.2 mod.	4 oz	Soil Jar	1	28 days (^)
Total Organic Halogen	9020/9021	4 oz	Soil Jar	1	28 days (^)
Total Petroleum Hydrocarbons-Extraction, IR	9071	4 oz	Soil Jar	1	28 days (^)
ELEMENTAL ANALYSES					
ICP Elements	6010	4 oz	Soil Jar	1	6 months
ICP MS ELements	6020	4 oz	Soil Jar	1	6 months
GFAA(Furnace) Elements	7000series	4 oz	Soil Jar	1	6 months
Mercury	7471	4 oz	Soil Jar	1	28 days

TABLE 1 (cont.)

SAMPLING AND PRESERVATION REQUIREMENTS

PARAMETER – SOLID MATRICES	METHOD	QUANTITY	CONTAINER	PRSV	HOLD TIME
ELEMENTAL ANALYSES **(cont.)**		**4 oz=100 g**			
Chromium, Hexavalent	3060/7196	4 oz	Soil Jar	1	30dys/24hrs
GC ORGANIC ANALYSES					
BTEX & MTBE	8021	(2) 40 mL	VOA Vial	1	14 days
Explosives - HPLC	8330, 8332	4 oz	Soil Jar	1	14days/40days
Extractable Petroleum Hydrocarbons	MADEP/EPH	4 oz	Soil Jar	1	7days/40days
Fuel Oil	ME HETL 4.1.25	4 oz	Soil Jar	1	14days/40days
Fule Oil	8015 mod.	4 oz	Soil Jar	1	14days/40days
Gasoline	ME HETL 4.2.17	(2) 40 mL	VOA Vial	1	14 days
Gasoline	8015 mod.	(2) 40 mL	VOA Vial	1	14 days
Herbicides	8151	4 oz	Soil Jar	1	14days/40days
PCB's (& Congeners)	8082	4 oz	Soil Jar	1	14days/40days
PCB's in Oil	8082	4 oz	VOA Vial	1	40 days
Pesticides	8081	4 oz	Soil Jar	1	14days/40days
Pesticides and PCB's	8081/8082	4 oz	Soil Jar	1	14days/40days
Purgeable Aromatics	8021	(2) 40 mL	VOA Vial	1	14 days
Purgeable Halocarbons	8021	(2) 40 mL	VOA Vial	1	14 days
Purgeables, Total	8021	(2) 40 mL	VOA Vial	1	14 days
Solvents (Direct Injection)	8015M	(2) 40 mL	VOA Vial	1	14 days
Volatile Petroleum Hydrocarbons	MADEP/VPH	(2)40 mL	VOA vial	13	28days
HPLC ANALYSES					
HPLC-Explosives	8330, 8332	4 oz	Soil Jar	1	7days/40days
GC/MS ANALYSES					
Acid Extractables-Priority Pollutants	8270	4 oz	Soil Jar	1	14 days/40 days
Acid Extractables-TCL	8270	4 oz	Soil Jar	1	14 days/40 days
Base Neutral Extractables-Priority Pollutants	8270	4 oz	Soil Jar	1	14 days/40 days
Base Neutral Extractables-TCL	8270	4 oz	Soil Jar	1	14 days/40 days
Polyaromatic Hydrocarbons	8270/8270SIM	4 oz	Soil Jar	1	14 days/40 days
Semivolatile Extractables-Priority Pollutants	8270	4 oz	Soil Jar	1	14 days/40 days
Semivolatile Extractables-TCL	8270	4 oz	Soil Jar	1	14 days/40 days
Volatile Organics – High Soil (>200 ug/kg)	5035/8260	Please refer to Table 6-2	Encore or similar sampler or VOA Vial or soil jar	14	Extruded w/in 48 hrs. Analyzed w/in 14 days
Volatile Organics – Low Soil (<200 ug/kg)	5035/8260	Please refer to Table 6-2	Encore or similar sampler or VOA Vial	14 or 15	Extruded w/in 48 hrs. Analyzed w/in 14 days
Volatile Organics-Priority Pollutants	8260	(2) 40 mL	VOA Vial	1	14 days
Volatile Organics-TCL	8260	(2) 40 mL	VOA Vial	1	14 days

TABLE 1 (cont.)

SAMPLING AND PRESERVATION REQUIREMENTS

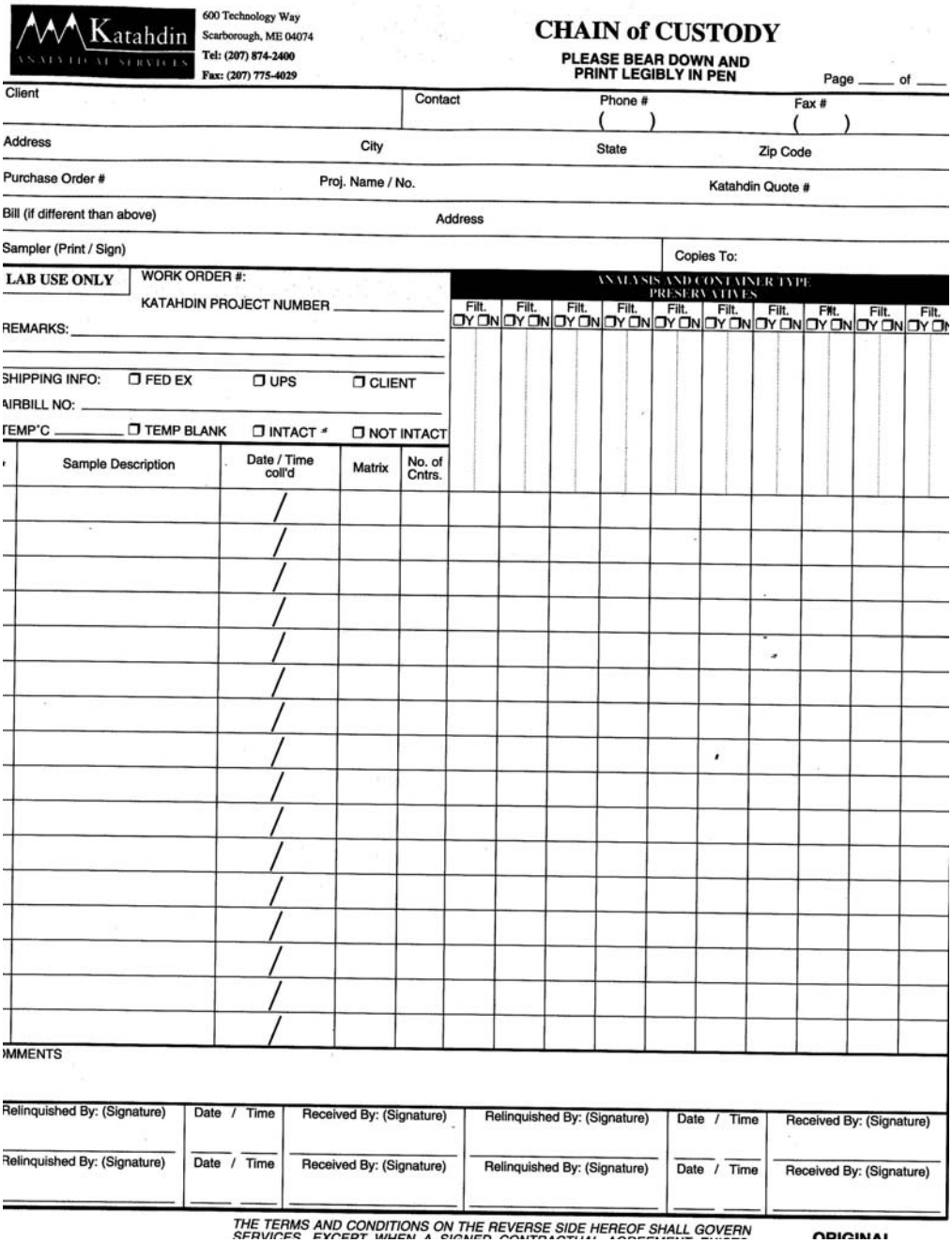
PARAMETER – SOLID MATRICES	METHOD	QUANTITY	CONTAINER	PRSV	HOLD TIME
RCRA - HAZARDOUS WASTE CHARAC.					
Corrosivity-pH	9045	4 oz	Soil Jar	1	24 hours (^)
Ignitability-Flash Point (closed cup)	1010	4 oz	Soil Jar	1	14 days (^)
Reactivity-Reactive Cyanide	7.3.3.2	4 oz	Soil Jar	1	14 days
Reactivity-Reactive Sulfide	7.3.4.1	4 oz	Soil Jar	1	7 days
TCLP					
TCLP Extraction-Volatile Organics	1311	100 g	Soil Jar	1	14 days
TCLP Extraction-Semivolatiles	1311	200 g	Soil Jar	1	14 days
TCLP Extraction-Pesticides & Herbicides	1311	400 g	Soil Jar	1	14 days
TCLP Extraction-Metals	1311	200 g	Soil Jar	1	28 days
TCLP Analysis-Volatile Organics	8260	see above	Soil Jar	1	14 days
TCLP Analysis-Metals	6010/6020	see above	Soil Jar	1	180 days
TCLP Analysis-Mercury	7470	see above	Soil Jar	1	28 days
TCLP Analysis-Semivolatiles	8270	see above	Soil Jar	1	7 days/40 days
TCLP Analysis-Pesticides	8081	see above	Soil Jar	1	7 days/40 days
TCLP Analysis-Herbicides	8151	see above	Soil Jar	1	7 days/40 days

METHODS OF PRESERVATION
1 = Cool at 4 Degrees Celsius
2 = Settled
3 = H2SO4 to pH<2
4 = HNO3 to pH<2
5 = NaOH to pH>12
6 = 1 mL 0.1M Na2S2O3 or 1 10 mg pellet
7 = 1 m/L 2NZnAc/L & NaOH
8 = 2 drops 1:1 HCl
9 = No headspace
10 = Na2S2O3, if chlorinated
11 = HCl to pH < 2
12 = 5 mL of HCL
13 = 15 mL of methanol
14 = methanol
15 = sodium bisulfate

TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

FIGURE 1

EXAMPLE OF STANDARD KATAHDIN CHAIN-OF-CUSTODY FORM



Katahdin
ANALYTICAL SERVICES
600 Technology Way
Scarborough, ME 04074
Tel: (207) 874-2400
Fax: (207) 775-4029

CHAIN of CUSTODY

PLEASE BEAR DOWN AND PRINT LEGIBLY IN PEN

Page ____ of ____

Client | Contact | Phone # ( ) | Fax # ( )

Address | City | State | Zip Code

Purchase Order # | Proj. Name / No. | Katahdin Quote #

Bill (if different than above) | Address

Sampler (Print / Sign) | Copies To:

LAB USE ONLY WORK ORDER #:

KATAHDIN PROJECT NUMBER _____

REMARKS: _____

SHIPPING INFO: ❒ FED EX ❒ UPS ❒ CLIENT

AIRBILL NO: _____

TEMP°C _____ ❒ TEMP BLANK ❒ INTACT ❒ NOT INTACT

ANALYSIS AND CONTAINER TYPE
PRESERVATIVES

Sample Description	Date / Time coll'd	Matrix	No. of Cntrs.	Filt. ❒Y ❒N	Filt. ❒Y ❒N	Filt. ❒Y ❒N	Filt. ❒Y ❒N	Filt. ❒Y ❒N	Filt. ❒Y ❒N	Filt. ❒Y ❒N	Filt. ❒Y ❒N	Filt. ❒Y ❒N	Filt. ❒Y ❒N
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COMMENTS

Relinquished By: (Signature)	Date / Time	Received By: (Signature)	Relinquished By: (Signature)	Date / Time	Received By: (Signature)
Relinquished By: (Signature)	Date / Time	Received By: (Signature)	Relinquished By: (Signature)	Date / Time	Received By: (Signature)

THE TERMS AND CONDITIONS ON THE REVERSE SIDE HEREOF SHALL GOVERN SERVICES, EXCEPT WHEN A SIGNED CONTRACTUAL AGREEMENT EXISTS

ORIGINAL

TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

FIGURE 2

EXAMPLE OF HOMEOWNER KATAHDIN CHAIN-OF-CUSTODY FORM

Katahdin
ANALYTICAL SERVICES

600 Technology Way
P.O. Box 540
Scarborough, ME 04070
Tel: (207) 874-2400 Fax: (207) 775-4029

Homeowner Chain of Custody

Client:	Contact:	Phone:	Fax:
Address:	City:	State:	Zip:
Purchase Order #:	Project Name/No.:	E-mail:	
Billing Address (if different):			
Sampler (Print/Sign):		Copies To:	

***Test results are for compliance and will be reported to the state (see statement below). yes no Compliance samples must be received on ice.

Lab Use Only | Work Order #: | KAS Project Manager:

Shipping: UPS Fed-Ex Mail Drop-Off

Sample(s) Received on Ice? Yes No Temperature if Iced:

Sample Description (Sample Identification and/or Lot #)	Date Collected	Time Collected	No. of Cntrs.	Standard Homeowner	Arsenic	Total Coliforms	Lead	Safety Test – coliform & N+N	FHA/MSH	Fluoride	Uranium		What's Included in the Standard Test and the FHA/MSH Test.
													Standard Homeowner Total Coliform/e-coli Nitrate, Nitrite Chloride, pH Hardness Copper, Iron Manganese Sodium
													FHA/MSH Standard plus Lead Turbidity Color Odor

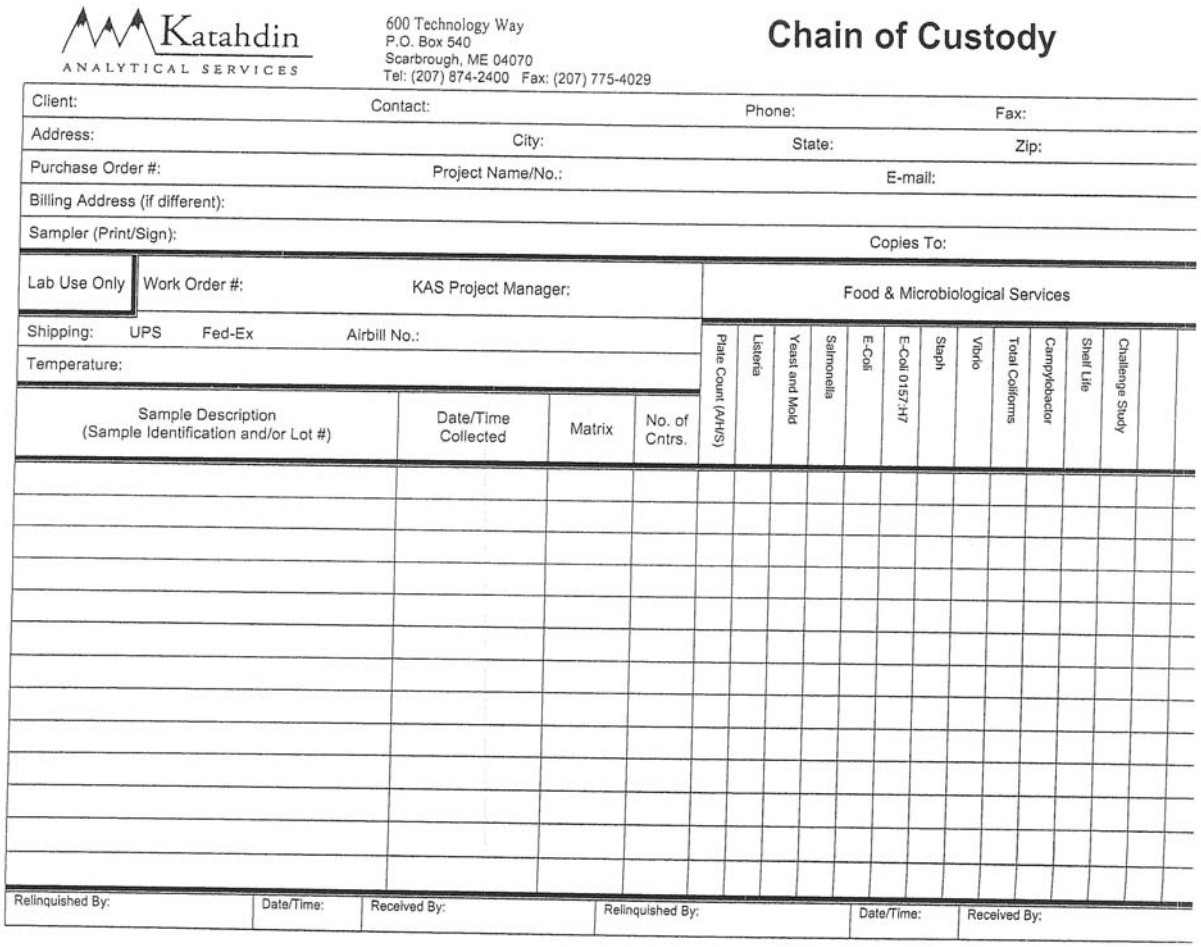
Relinquished By:	Date/Time:	Received By:	Relinquished By:	Date/Time:	Received By:

Per the National Environmental Laboratory Accreditation Program (NELAP) Standards, Katahdin is required to accept samples that have been properly preserved. All sample containers provided to you have been properly preserved, but the proper preservation also requires samples to be received at <6 degrees celcius. The Safe Drinking Water Act regulations only require this for compliance samples (i.e., results that are submitted to the state). By circling no for compliance (above), you acknowledge that the samples described above are not for compliance purposes, and thus may not meet the temperature receipt requirements.

TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

FIGURE 3

EXAMPLE OF FOOD/MICROBIOLOGY KATAHDIN CHAIN-OF-CUSTODY FORM



Katahdin
ANALYTICAL SERVICES

600 Technology Way
P.O. Box 540
Scarbrough, ME 04070
Tel: (207) 874-2400 Fax: (207) 775-4029

Chain of Custody

Client: Contact: Phone: Fax:

Address: City: State: Zip:

Purchase Order #: Project Name/No.: E-mail:

Billing Address (if different):

Sampler (Print/Sign): Copies To:

Lab Use Only | Work Order #: KAS Project Manager: Food & Microbiological Services

Shipping: UPS Fed-Ex Airbill No.:

Temperature:

Sample Description (Sample Identification and/or Lot #)	Date/Time Collected	Matrix	No. of Cntrs.	Plate Count (AHPS)	Listeria	Yeast and Mold	Salmonella	E-Coli	E-Coli 0157:H7	Staph	Vibrio	Total Coliforms	Campylobacter	Shelf Life	Challenge Study		

Relinquished By:	Date/Time:	Received By:	Relinquished By:	Date/Time:	Received By:

TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

FIGURE 4

EXAMPLE OF KATAHDIN SAMPLE RECEIPT LOGBBOK

KATAHDIN ANALYTICAL SERVICES, INC.

SAMPLE LOG IN

Date Received	Time Received	Date Logged In	Time Logged In	Work Order	Client	Initials
				SA 0094		
				SA 0095		
				SA 0096		
				SA 0097		
				SA 0098		
				SA 0099		
				SA 0100		
				SA 0101		
				SA 0102		
				SA 0103		
				SA 0104		
				SA 0105		
				SA 0106		
				SA 0107		
				SA 0108		
				SA 0109		
				SA 0110		
				SA 0111		
				SA 0112		
				SA 0113		
				SA 0114		
				SA 0115		
				SA 0116		
				SA 0117		
				SA 0118		
				SA 0119		
				SA 0120		
				SA 0121		
				SA 0122		
				SA 0123		
				SA 0124		

Signed By:_____ Date :_____

Reviewed By:_____ Date:_____

0000004

TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

FIGURE 5

EXAMPLE OF WET CHEMISTRY SHORTS AND RUSHES LOGBOOK

WET CHEMISTRY SHORTS & RUSHES **Receipt Date:**

HOLDING TIME					Immediate			24 Hr		48 Hr								
Work Order / Client	Matrix	Earliest Sampling Date	Earliest Sampling Time	Rush Parameters	pH	DO	Sulfite	Fe +2	Cr+6	Total BOD	Carbon. BOD	Color	Nitrate	Nitrite	OPO4	Set Solids	Turbidity	Comments (Quick TAT, MS/MSD, etc.)

000004

TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

FIGURE 6

EXAMPLE OF SAMPLE RECEIPT CONDITION REPORT FORM

Katahdin Analytical Services, Inc. **Sample Receipt Condition Report**

Client:	KAS PM:	Sampled By:
Project:	KIMS Entry By:	Delivered By:
KAS Work Order#:	KIMS Review By:	Received By:
SDG #:	Cooler: _____ of _____	Date/Time Rec.:

Receipt Criteria	Y	N	EX*	NA	Comments and/or Resolution
1. Custody seals present / intact?					
2. Chain of Custody present in cooler?					
3. Chain of Custody signed by client?					
4. Chain of Custody matches samples?					
5. Temperature Blanks present? If not, take temperature of any sample w/ IR gun.					Temp (°C):
Samples received at <6 °C w/o freezing?					Note: Not required for metals analysis.
Ice packs or ice present?					The lack of ice or ice packs (i.e. no attempt to begin cooling process) may not meet certain regulatory requirements and may invalidate certain data.
If not, has the cooling process begun (i.e. ice or packs present) and sample collection times <2hrs., but samples are not yet cool?					Note: No cooling process required for metals analysis.
6. Volatiles free of headspace: **Aqueous:** No bubble larger than a pea **Soil/Sediment:** Received in airtight container?					
Received in methanol?					
Methanol covering soil?					
7. Trip Blank present in cooler?					
8. Proper sample containers and volume?					
9. Samples within hold time upon receipt?					
10. Aqueous samples properly preserved? Metals, COD, NH3, TKN, O/G, phenol, TPO4, N+N, TOC, DRO, TPH – pH <2 Sulfide - >9 Cyanide – pH >12					

* Log-In Notes to Exceptions: document any problems with samples or discrepancies or pH adjustments

TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

FIGURE 7

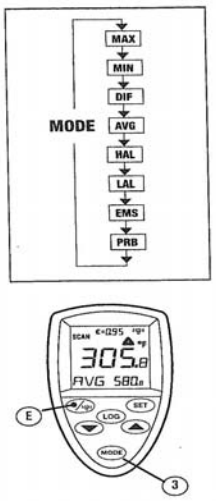
IR THERMOMETER MANUFACTURER'S INSTRUCTIONS FOR CHANGING EMISSIVITY

MODE Button Functions

Your infrared thermometer measures Maximum (MAX), Minimum (MIN), Differential (DIF)*, and Average (AVG)** temperatures each time you take a reading. This data is stored and can be recalled with the MODE button (3) until a new measurement is taken. (See "Hold and Recall" for information on how to recall stored data.) When the trigger is pulled again, the unit will begin measuring in the last mode selected. Pressing the MODE button also allows you to access the High Alarm (HAL), Low Alarm (LAL), Emissivity (EMS), Probe temperature (PRB—only available when the probe is connected), and Data logger (LOG). Each time you press MODE, you advance through the mode cycle. The diagram shows the sequence of functions in the Mode cycle.

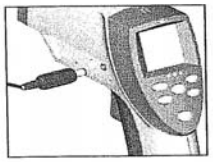
Note: PRB (probe) is only available in the MODE loop when the contact probe is connected to the unit.

*DIF shows the difference between the maximum and minimum temperatures measured.
**AVG shows the average temperature reading for each time the trigger is pulled or the unit is locked on.



Selecting a Function

To Select the MAX, MIN, DIF, or AVG mode, pull the trigger. While holding the trigger, press the MODE button (3) until the appropriate code appears in the lower left corner of the display (E). Each time you press MODE, you advance through the MODE cycle. The MODE cycle is shown above.



Setting the High Alarm, Low Alarm, and Emissivity

To set values for the High Alarm (HAL), Low Alarm (LAL), and Emissivity, pull the trigger or press the MODE button (3) to activate the display. Press the MODE button until the appropriate code appears in the lower left corner of the display (E). Use the up and down keys (2) to adjust the desired values. To activate the alarms, press SET (1). To deactivate the alarms, press SET again.

Using a Probe (PRB)

Connect the probe to the input on the side of the unit (as shown). PRB automatically appears in the lower left corner of the display (E, below). The probe temperature is shown in the lower right part of the display. The current infrared temperature continues to show in the center of the display (F). While the probe is connected, you may still cycle through the mode functions by pressing MODE (3).

Note: PRB is only available in the MODE loop when a probe is connected to the unit; the probe temperature will not activate the high alarm or low alarm.

TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

FIGURE 8

EXAMPLE OF KATAHDIN SAMPLE FILTRATION LOGBOOK

KATAHDIN ANALYTICAL SERVICES, INC.

Sample Filtration Logbook

Katahdin Sample No. List Individually	Site ID (Optional)	Filtration Requested By: Initials	Date	Filtered and Preserved By: Initials	Date	Time

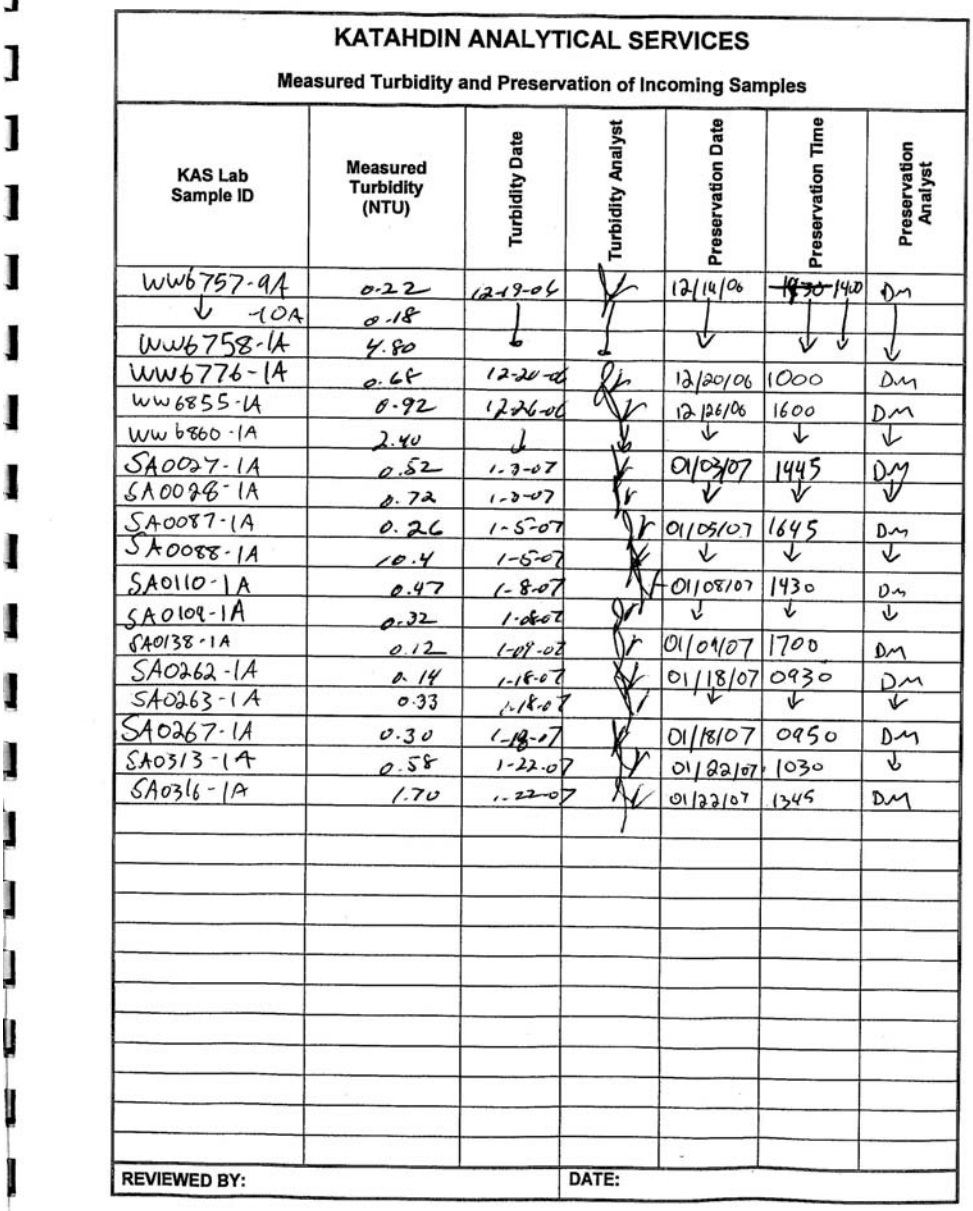
Reviewed and Approved by: _____ Date: _____

QAAA097 0000001

TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

FIGURE 9

MEASURED TURBIDITY AND PRESERVATION OF INCOMING SAMPLES LOGBOOK



KATAHDIN ANALYTICAL SERVICES

Measured Turbidity and Preservation of Incoming Samples

KAS Lab Sample ID	Measured Turbidity (NTU)	Turbidity Date	Turbidity Analyst	Preservation Date	Preservation Time	Preservation Analyst
WW6757-9A	0.22	12-19-06		12/14/06	~~1430~~ 1440	DM
↓ 10A	0.18	↓		↓	↓ ↓	↓
WW6758-1A	4.80	↓		↓	↓ ↓	↓
WW6776-1A	0.68	12-20-06		12/20/06	1000	DM
WW6855-1A	0.92	12-26-06		12/26/06	1600	DM
WW6860-1A	2.40	↓		↓	↓	↓
SA0027-1A	0.52	1-3-07		01/03/07	1445	DM
SA0028-1A	0.72	1-3-07		↓	↓	↓
SA0087-1A	0.26	1-5-07		01/05/07	1645	DM
SA0088-1A	0.4	1-5-07		↓	↓	↓
SA0110-1A	0.47	1-8-07		01/08/07	1430	DM
SA0109-1A	0.32	1-8-07		↓	↓	↓
SA0138-1A	0.12	1-09-07		01/09/07	1700	DM
SA0262-1A	0.14	1-18-07		01/18/07	0930	DM
SA0263-1A	0.33	1-18-07		↓	↓	↓
SA0267-1A	0.30	1-18-07		01/18/07	0950	DM
SA0313-1A	0.58	1-22-07		01/22/07	1030	↓
SA0316-1A	1.70	1-22-07		01/22/07	1345	DM

REVIEWED BY: **DATE:**

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FIGURE 6

TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

FIGURE 10

EXAMPLE OF LABORATORY INCOMING SAMPLE INFORMATION SHEET (ISIS)

KATAHDIN ANALYTICAL SERVICES, INC. – INCOMING SAMPLE INFORMATION SHEET

CLIENT:	ATTENTION:
ADDRESS:	
TELEPHONE:	FAX:
OTHER CLIENT CONTACTS:	
REPORT CC:	PM/SALES CONTACT:
PO #:	CLIENT ACCOUNT:
KAS QUOTE #:	KIMS QUOTE #:
PROJECT NAME:	KIMS PROJECT REF.:
SDG:	APPLICABLE CERTIFICATION:
RECEIPT DATE:	MEANS OF DELIVERY:

METHOD OR KIMS PRODUCT	MATRIX	# OF SAMPLES	SPECIAL INSTRUCTIONS (I.E LIMITS, PREPS)

SUBCONTRACT PARAMETER	SUBCONTRACT LABORATORY

QC LEVEL:	EDD:
RUSH/VERBAL TAT:	HARDCOPY TAT:
HISTORY:	
OTHER:	

TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

FIGURE 11

EXAMPLE OF KATAHDIN WORK ORDER/LOGIN COC REPORT

Katahdin
ANALYTICAL SERVICES

Katahdin Analytical Services

Login Chain of Custody Report (lno1)

Jan. 26, 2007
03:51 PM

Page: 1 of 1

Login Number: SA0395
Account: KATAHD001 Web
Katahdin Analytical Services
Project:

Primary Report Address:
Leslie Dimond
Katahdin Analytical Services
600 Technology Way
P.O. Box 540
Scarborough,ME 04070

Primary Invoice Address:
Accounts Payable
Katahdin Analytical Services
600 Technology Way
P.O. Box 540
Scarborough,ME 04070

Report CC Addresses:
Invoice CC Addresses:

Login Information

ANALYSIS INSTRUCTIONS	:
CHECK NO.	:
CLIENT PO#	:
COOLER TEMPERATURE	: n/a
DELIVERY SERVICES	: In House
EDD FORMAT	:
MAIL DATE	:
PM	: LAD
PROJECT NAME	: QC Holding Blanks
QC LEVEL	: I
REGULATORY LIST	:
REPORT INSTRUCTIONS	:
SDG ID	:
SDG STATUS	:

Laboratory Sample ID	Client Sample Number	Collect Date/Time	Receive Date	PR	Verbal Date	Due Date	Comments
SA0395-1	WHITE FRIDGE	26-JAN-07 15:50	26-JAN-07			08-FEB-07	

Matrix		Product	Hold Date (shortest)	Bottle Type	Bottle Count
Aqueous	S	SW8260FULL5ML	09-FEB-07		2

Laboratory Sample ID	Client Sample Number	Collect Date/Time	Receive Date	PR	Verbal Date	Due Date	Comments
SA0395-2	BLUE FRIDGE	26-JAN-07 15:50	26-JAN-07			08-FEB-07	

Matrix		Product	Hold Date (shortest)	Bottle Type	Bottle Count
Aqueous	S	SW8260FULL5ML	09-FEB-07		2

Total Samples: **2** ***Total Analyses:*** **2**

TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

FIGURE 12

EXAMPLE OF LABORATORY INTERNAL CHAIN-OF-CUSTODY FORM

Katahdin Analytical Services, Inc.
Internal Custody Record

EXTRACTIONS (AQ) Work Order #:_____

Sample Numbers Involved	Placed in refrigeration by: Initials
	Date
	Time

Sample Numbers	Analysis	Removed			Returned			Consumed?	
		Initials	Date	Time	Initials	Date	Time	Yes	No

TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

FIGURE 13

EXAMPLE OF REFRIGERATOR TEMPERATURE LOGBOOK

KATAHDIN ANALYTICAL SERVICES, INC.

SAMPLE RECEIPT WALK-IN TEMPERATURE LOG

Corrective Action: Note in the "comments" column and notify the QAO or supervisor; document corrective actions taken and return to control.

Thermometer Location		Sample Receipt Walk-in 1	Comments
Acceptance Criteria		2 to 6 °C	
Date	Initials	Temp (°C)	
02/2/07	NDA	3.7	
02/05/07	DWM	5.8	
02/06/07	DWM	5.8	
02/07/07	DWM	5.8	
02/08/07	DWM	3.8	
02/09/07	DWM	3.5	
02/12/07	DWM	3.5	
02/13/07	DWM	3.5	

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TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

FIGURE 14

EXAMPLE OF IMMEDIATE INTERNAL COC LOGBOOK

KATAHDIN ANALYTICAL SERVICES, INC.
INTERNAL CUSTODY RECORD FOR IMMEDIATES

CLIENT	PROJECT	CLIENT ID &/or WORK ORDER #	ANALYSIS	OUT date/time	IN date/time	INIT	Consumed?
Jacobs		WW4813-1A, -2A	ICP	9/13/06 0930	→0935	DJJ	yes no
Jacobs		WW4883-1A	ICP	9/16/06 0900	→1000	DJJ	yes no
CES		WW4965	BOD	9/20/06 0830	9/20/06 1000	CP	yes no
CCAR		WW4969	BOD	9/20/06 1000	↓	CP	yes no
GEMF		WW4970	BOD	↓	↓	CP	yes no
Jacobs		WW4962-1A, -2A	ICP	9/20/06 0900	→1000	DJJ	yes no
Irving		WW4994	BOD	9/21/06 1000	9/21/06 1045	CP	yes no
Highliner		WW4992 ②	BOD	9/21/06 1015		CP	yes no
NATIONAL		WW5000	TS, PEROXIDE PH, SP. COND	9/21/06 1100	9/21/06 1257	DJJ	yes no
WTC		WW5001	BOD	9/21/06 1300		CP	yes no
Allens		WW5016	NO3	9/22/06 1300	9/22/06 1100	KP	yes no
RANSOM		WW5010	↓	↓	↓	↓	yes no
EcoMaine		WW5029	BOD	9/20/06 1100		CP	yes no

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KATAHDIN ANALYTICAL SERVICES, INC.
STANDARD OPERATING PROCEDURE

SOP Number: SD-903
Revision History
Cover Page
Page 1

TITLE: **SAMPLE DISPOSAL**

Prepared By: _____ Date: 2/01

Approved By:

Group Supervisor: _____ Date: _____

Operations Manager: Jal C. Burton Date: 2/01

QA Officer: Deborah J. Nadeau Date: 2.01

General Manager: _____ Date: 2/01

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
01	Format changes, ~~added pollution prevention~~, added updated log-book and greater detail on disposal.	DN	2.01	2/01
02	Major rewrite to include more detail on hazardous waste regulations & to reflect current practices.	DN	02/05	02/05
03	Rewrite of section 7 to comply with current practices in new facility. Updated Figures 1 to 3.	DN	02.08	02.08
04	Added elementary neutralization to section 7.0. Other minor edits.	DN	05.09	0509

TITLE: SAMPLE DISPOSAL

Please acknowledge receipt of this standard operating procedure by signing and dating both of the spaces provided. Return the bottom half of this sheet to the QA Department.

I acknowledge receipt of copy ___ of document **SD-903-04**, titled **SAMPLE DISPOSAL**.

Recipient: _____ Date: _____

KATAHDIN ANALYTICAL SERVICES, INC.
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy ___ of document **SD-903-04**, titled **SAMPLE DISPOSAL**.

Recipient: _____ Date: _____

TITLE: SAMPLE DISPOSAL

1.0 SCOPE AND APPLICATION

Katahdin Analytical Services, Inc. requires strict adherence to specific procedures for the disposal of samples. The procedures are designed to categorize waste materials, provide for their safe and timely disposal and to ensure compliance with local and federal regulations pertaining to disposal of chemicals and environmental samples. Any other means of disposal not described in this SOP is prohibited without consent from the Katahdin Environmental Health & Safety Officer and/or the Katahdin Environmental Compliance Officer.

The purpose of this SOP is to describe the procedures utilized by Katahdin Analytical personnel for the disposal of samples. These procedures apply to the disposal of all samples received or processed by Katahdin. Refer to the current revision of Katahdin SOP CA-107 regarding the disposal of spent preparation and analysis reagents, standards, sample extracts, distillates, or digestates.

1.1 Definitions

Hazardous Waste – A “Solid Waste” which displays a hazardous characteristic or is specifically listed as hazardous waste.

Solid Waste – Any discarded material that is not excluded from the definition of hazardous waste.

Discarded Material – Material that is abandoned, recycled or inherently waste-like.

Waste (State of Maine) –

- Any useless, unwanted, or discarded substance or material, whether or not such substance or material has any other future use.
- Any substance or material that is spilled, leaked, pumped, poured, emptied or dumped onto the land or into the water or ambient air.
- Materials which are used in a matter constituting disposal, burned for energy recovery, reclaimed, or accumulated speculatively.

Ignitable Hazardous Waste – EPA Waste Code D001

- Liquids with a flash point less than 140°F or 60°C.
- Solids capable of spontaneous combustion under normal temperature and pressure.
- Ignitable compressed gas.
- Oxidizers.

Corrosive Hazardous Waste - Liquids with a pH less than or equal to 2.0 or greater than or equal to 12.5. EPA waste code D002.

Reactive Hazardous Waste – EPA waste code D003.

- A material that reacts violently with water.
- A material that generates toxic gases or fumes.
- Explosives.

Toxic Hazardous Waste – A material that exceeds certain concentration levels based on the toxicity characteristic leaching procedure (TCLP). See Figure 3 for the chemicals and concentration levels covered under this definition.

Listed Wastes – Lists of chemicals that are considered hazardous based on the following criteria

- Virgin chemical or unused product.
- Sole active ingredient.
- Single substance spill debris.

Listed wastes are divided into 5 subcategories

- F-wastes – Describe hazardous waste from non-specific sources usually containing halogenated and non-halogenated solvents.
- K-wastes – Describe hazardous wastes created by specific processes.
- U-wastes – Describe toxic or non-acute hazardous wastes.
- P-wastes – Describe acute hazardous wastes. (Note: Maine considers a material to be a P-listed waste if it contains 10% or more of any P-listed chemical.
- State listed wastes – Maine lists any material with a concentration of greater than 50 ppm Polychlorinated Biphenyls (PCB) as a hazardous waste.

Organics hit – A liquid sample containing greater than 1 mg/L of organic contaminants or a soil sample containing greater than 20 mg/kg of organic contaminants.

1.2 Responsibilities

Only designated analysts/technicians trai ned in these procedures may dispose of samples or analytical by-products. Each analyst or technician must be familiar with Katahdin Analytical safety procedures. Gloves, safety glasses, lab coats and/or other protective clothing must be worn at all times.

It is the responsibility of the designated Katahdin personnel involved in the disposal of samples to read and understand this SOP, to adhere to the procedures outlined, to properly document their activities in the appropriate lab notebook and file the necessary manifests and reports to outside agencies in the required manner. Refer to

Katahdin SOP QA-805, "Personnel Training & Documentation of Capability," current revision.

It is the responsibility of the Department Managers to oversee that members of their group follow this SOP, to ensure that their work is properly documented and to initiate periodic review of the associated logbooks.

It is the responsibility of the Katahdin Environmental Health & Safety Officer (EHSO) to manage the proper classification and disposal of samples. Katahdin is responsible for regulatory compliance of Katahdin's waste storage areas (less than 90 day storage). The EHSO ensures compliance of the waste storage areas with applicable state and federal regulations. The EHSO is responsible for providing the appropriate training to all individuals involved in the proper classification and/or disposal of samples. The EHSO is responsible for working with the Laboratory Operations Manager/Environmental Compliance Officer to help identify problems and assure resolution, to facilitate corrective action where needed, and to communicate unresolved problems and concerns to the Laboratory Vice President.

It is the responsibility of the Operations Manager/Environmental Compliance Officer to oversee adherence to Katahdin sample disposal and hazardous waste practices by all laboratory groups under his/her authority, to help identify problems and assure resolution, to facilitate corrective action where needed, and to communicate problems and concerns to the EHSO and/or the Laboratory Vice President.

It is the responsibility of the Laboratory Vice President to provide the necessary resources to meet the regulatory requirements of proper classification and disposal of samples.

2.0 SUMMARY OF METHOD

Not applicable.

3.0 INTERFERENCES

Not applicable.

4.0 APPARATUS AND MATERIALS

Not applicable.

TITLE: SAMPLE DISPOSAL

5.0 REAGENTS

Not applicable.

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

Not applicable.

7.0 PROCEDURES

7.1 Sample purging is the removal of samples from laboratory refrigerated storage. Sample storage areas where samples are removed (purged) from include wet chemistry, organic extractables, metals, volatiles, total organic carbon and soils. Wet chemistry, aqueous metals, organic extractables, total organic carbon, and soils can all be found in the walk-in refrigerator. Aqueous and soil volatiles can be found in the volatiles laboratory refrigerators/freezer.

7.2 Samples are purged from storage, after analysis and reporting, on a routine basis to make room for incoming samples. Samples are to be kept in storage for a duration of 30 days past the report mailed date. Some samples must be kept for 60 or 90 days beyond the report mailed date, depending on specific client requests and contracts.

7.3 The first step in disposing of samples is to generate a disposal list. The disposal list contains sample analysis information stored in the Katahdin Information Management System (KIMS). The analytical data for the samples is compared to the hazardous waste criteria specified in 40CFR Part 261 and to local wastewater discharge criteria. Refer to Figure 4 for 40 CFR Part 261 Characteristic Hazardous Waste Criteria. Based on this comparison, the report displays information on the classification/category for disposal of each sample. The disposal report should be reviewed against the data reports for accuracy. Refer to Figure 2 for an example of a KIMS generated disposal list. The primary disposal categories listed in the report are: non-hazardous, high organics, high metals, flashpoint, high mercury, high PCBs, and high cyanide. K atahdin has established 14 waste stream profiles with a 3rd party waste transporter/waste disposal firm for sample disposal based on these categories. As required, new or special temporary waste profiles are established based on the characteristics of samples.

7.4 Sorting through samples and preparing them for disposal is a crucial quality checkpoint. Samples put into the incorrect waste stream could not only produce adverse environmental effects, but, could also interrupt the 3rd party's waste treatment efficiency, or endanger an individual handling the waste stream. Therefore, when sorting through samples pay close attention to which waste stream each sample falls into.

7.5 Once you are ready to dispose of the samples of interest (the oldest samples that have been purged), these sam ples must be sorted, logged, and the classification/category (sample knowledge) information recorded.

Sample storage times (as listed in section 7.2) and space should be taken into consideration when purging samples. It is important to make room for future samples, but to make sure that samples are not purged too early. Samples should be pulled from the walk-in or the volatiles refrigerators to make room for new samples. When purging, chose a section that needs extra space the most and remove the oldest samples.

Safety glasses, nitrile gloves, lab coat, and a splash apron must be worn when handing samples during disposal

7.6 Remove the designated purge samples from the shelf one by one and line them up on the countertop in the log-in area. Generally, removing two cartloads at a time is a good amount to purge at one time. For volatile samples in 40mL vials, 5 or 6 vial trays should be purged at a time. Samples should be lined up across the counter with the earliest sample to the left and building up to the right, organizing the samples according to work order and sample number. After the samples are lined up, they should be recorded in the Sample Disposal Logbook (SDL). Refer to Figure 1 for an example SDL page. The location the samples were removed from should also be recorded. Sample storage areas are recorded with the following designations:

VOA	(Aq)	Aqueous Volatiles(VOA)
VOA	(SL)	Solid Volatiles(VOA)
M		Metals
EX	T	Extractables (Organic)
TOC		Total Organic Carbon
WC		Wet Chemistry
S		Soils

7.7 The next step is to use the sample disposal list to determine the earliest release date of the reports and to determine each samples appropriate waste classification/characterization. As stated in section 7.3, the primary disposal categories listed in the report are: non-hazardous, high organics, high metals, flashpoint, high mercury, high PCBs, and high cyanide.

Using the information from the KIMS disposal list, record the appropriate classification for each sample in the SDL. If multiple categories are identified as being present then a single category is selected as controlling. The order of precedence is PCB's, metals and then organics. If another scenario is found, the individual should bring it to the EHSO for a determination of the acceptable waste stream designation or a determination that it should be lab packed separately.

If samples have been sorted that have not been in storage for the 30 days beyond the release date (60 or 90 for certain clients), then these samples need to be placed back in storage and it should be noted in the SDL.

7.8 As stated above, a sample may be categorized into a waste stream based upon the analytes it contains as determined by laboratory testing. In addition, many samples are also categorized as hazardous waste based upon the preservative that they contain. Since many samples contain preservatives, caution must be used when dumping samples. It is also important to ensure that the sample container is empty. This can be accomplished by holding the container upside down and shaking gently until liquid is no longer observed coming out of the container.

7.9 Once waste categories have been determined and entered into the SDL, The following waste categories are disposed of as follows:

7.9.1 Dumping non-hazardous samples (as determined by laboratory testing)

Non-hazardous samples (non-preserved) are poured directly into the sink in the warehouse.

Non-hazardous solid samples are disposed of with the general trash, which is picked up by commercial trash collectors and ultimately disposed of in a waste-to-energy incinerator.

Sample containers from non-hazardous samples are disposed of with the general trash.

7.9.2 Dumping Samples with high Organics (as determined by laboratory testing)

Aqueous samples get dumped into waste stream “K”. Containers are disposed of with general trash. Solid samples are placed into waste stream “I” with their containers. The disposal date is recorded in the SDL.

7.9.3 Dumping samples high in metals, including mercury (as determined by the by laboratory testing)

Aqueous samples get disposed of in waste stream “A”. Containers are disposed of with general trash. Solid samples are placed in waste stream “L” with their containers. The disposal date is recorded in the SDL.

7.9.4 Dumping Acidic Samples that do not contain any other hazardous waste constituents (as determined by the acidic preservative or by laboratory testing)

Refer to section 7.10 below.

7.9.5 Dumping Basic samples (as determined by the basic preservative or by laboratory testing)

Aqueous samples get disposed of in waste stream “NHi”. Containers are disposed of with general trash. The disposal date is recorded in the SDL.

7.9.6 Dumping samples with high PCBs (as determined by laboratory testing)

Aqueous samples are disposed of in waste stream “Q”. Containers are disposed of with general trash. Solid samples get disposed of in waste stream “F” with their containers. The disposal date is recorded in the SDL.

7.9.7 Dumping samples with low flashpoints (as determined by laboratory testing)

Aqueous samples are disposed of in waste stream “O”. Containers are disposed of with general trash. Solid samples get disposed of in waste stream “I” with their containers. The disposal date is recorded in the SDL.

7.9.8 Dumping samples with high cyanide (as determined by laboratory testing)

Aqueous samples are disposed of in waste stream “NHi”. C ontainers are disposed of with general trash. Solid samples should be set aside for labpack. The disposal date is recorded in the SDL.

7.9.9 Miscellaneous Disposal (as determined by the preservative)

Sodium Bisulfate: Sodium Bisulfate often comes in vials, but may also come in the 2-4oz glass jars. Dump the Sodium Bisulfate out of the container into waste stream “A”. There may be remaining soil left in the sample container. The soil’s waste stream and dump date will be dictated by the SDL. The disposal date is recorded in the SDL.

Methanol / Free Products: This often comes in vials, but may also come in the 2-4oz glass jars. Dump the methanol out of the container into the mix-flammables accumulation. When this satellite accumulation container gets full it can be dumped into the “O” waste stream. There may be remaining soil left in the sample container. The soil’s waste stream and dump date will be dictated by the SDL. Lastly, samples marked “free product” on the Katahdin sample ID label can be dumped into the mixed flammables stream. The disposal date is recorded in the SDL.

7.10 Pursuant to Maine DEP regulations, Katahdin has the necessary agreements, processes and documentation in place to neutralize samples without a license. Refer to the current revision of the Katahdin Environmental Health & Safety Manual for additional information. Generally, the following procedures are followed.

7.10.1 Samples that have been determined to be hazardous due **solely** to the corrosivity characteristic are neutralized using sodium hydroxide pellets. In the warehouse, samples are emptied into a five gallon heavy duty carboy to about 60% capacity. The carboy is kept in a secondary container. Sodium hydroxide pellets are added slowly to t he carboy (about 5 grams at a time) and stirred with a long glass stirring rod. The pH is checked with pH paper.

7.10.2 This process is continued until the pH is between 7 and 8. This normally takes about 30-40 grams of sodium hydroxide pellets, but may vary depending on the buffering capacity of the individual samples.

7.10.3 The carboy is emptied into the sink in the warehouse. The tap water is run at the same time as the neutralized material is disposed of. An eyewash station and spill material is located at this sink.

7.10.4 All neutralization activities are documented, including the date and time of neutralization, the name of the person doing the neutralizing, the amount of neutralized liquid discharged, details on the inspection of the drain area and the date and nature of any significant repairs or corrective actions. This documentation is maintained by the EHSO. Refer to Figure 5 for an example logbook page of neutralization documentation.

7.11 Every 3 to 5 weeks a pickup of hazardous waste is scheduled with the 3rd party waste transporter/waste disposal firm. An inventory is faxed to the transporter summarizing the number of drums and waste streams/profiles. As required, a “lab pack” of expired chemicals or orphan samples is organized as necessary. A designated individual, with applicable Hazardous Waste (RCRA) and Department of Transportation (DOT) training, oversees the waste pickup and signs the hazardous manifests and land ban documentation. Within 7 days a copy is forwarded to the Maine Department of Environmental Protection (MEDEP) and the environmental agency in the designation state (if required by that state). Once the report is received at the disposal facility a copy is returned to KATAHDIN and the MEDEP.

7.12 Prior to March 31 of each year, the laboratory prepares the Annual Hazardous Waste Report (i.e., MEDEP modified EPA Form 8700-13A) as required by MEDEP Hazardous Waste Management Rules. The complete report is reviewed by the Katahdin Environmental Compliance Officer and then forwarded to the following address:

Maine Department of Environmental Protection
Bureau of Remediation & Waste Management
State House Station #17
Augusta, ME. 04333
Attn: Annual Hazardous Waste Report

TITLE: SAMPLE DISPOSAL

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

On a daily basis, a designated individual performs quality checks in all hazardous waste storage areas. The daily check documentation is located in login. Any discrepancy is copied to the Operations Manager and the Katahdin Vice President for corrective action. Refer to the current revision of Katahdin SOP CA-107, *The Management of Hazardous Waste as it Relates to the Disposal of Laboratory Process Waste, Reagents, Solvents & Standards*, for more information. Refer to Figure 3 for a copy of the daily check documentation.

9.0 METHOD PERFORMANCE

Not applicable.

10.0 APPLICABLE DOCUMENTS/REFERENCES

USEPA Code of Federal Regulations, 40 CFR Part 261.

Maine Department of Environmental Protection (ME DEP) Hazardous Waste Management Rules

ME DEP modified EPA Form 8700-13A

LIST OF TABLES AND FIGURES

TITLE: SAMPLE DISPOSAL

FIGURE 1

EXAMPLE OF SAMPLE DISPOSAL LOGBOOK (SDL)

KATAHDIN ANALYTICAL SERVICES, INC. –SAMPLE STORAGE/DISPOSAL LOGBOOK

WORK ORDER/ SAMPLE NUMBERS	DEPARTMENT	EARLIEST RELEASE DATE	< CRITERIA	SAMPLE KNOWLEDGE									DATE DISPOSED	INITIALS
			CLEAN	WL	ORG	METS	CN	FP	HG	PCBS				
SAS783-1	WC	10-17-07	✓										1-22-08	GN
SAS786-1		10-17-07	✓											
SAS787-1,2,4		10-17-07	✓											
SAS790-1		10-14-07		✓										
SAS793-1		10-17-07	✓											
SAS795-1-9		10-23-07	✓											
SAS797-1		10-23-07	✓											
SAS798-1,2		10-25-07			✓									
SAS799-1-5		10-31-07	✓											
SAS804-1,2		10-25-07	✓											
SAS809-1,2		10-23-07	1			2				2				
SAS810-1-4	↓	10-23-07	✓										↓	

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TITLE: SAMPLE DISPOSAL

FIGURE 2

EXAMPLE OF KIMS GENERATED WASTE DISPOSAL REPORT

SAMPLE DISPOSAL REPORT

Query by: Login SA6501 to SA7000
Date : 15-JAN-08

Sample	SDG	Status	Mail Date	Parameter	Value		
SA6605-1		NEED	12/02/07				
SA6606-1		NEED	12/02/07				
SA6607-1		NEED	11/15/07				
SA6608-1		NEED	12/06/07				
				ORG	1.17	MG/L	(HIGH)
SA6608-1		NEED	12/06/07				
SA6608-2		NEED	12/06/07				
				AA	13	MG/KG	(HIGH)
SA6609-1		NEED	11/26/07				
SA6609-1		NEED	11/26/07				
SA6610-1		NEED	11/30/07				
SA6611-1	FCS-020	NEED	12/07/07				
SA6611-2	FCS-020	NEED	12/07/07				
SA6611-3	FCS-020	NEED	12/07/07				
SA6611-4	FCS-020	NEED	12/07/07				
SA6611-5	FCS-020	NEED	12/07/07				
SA6611-6	FCS-020	NEED	12/07/07				
SA6611-7	FCS-020	NEED	12/07/07				
SA6611-8	FCS-020	NEED	12/07/07				
SA6612-1	NSA-030	NEED	12/07/07				
SA6612-2	NSA-030	NEED	12/07/07				
SA6612-3	NSA-030	NEED	12/07/07				
SA6612-4	NSA-030	NEED	12/07/07				
				ORG	1.70735	MG/L	(HIGH)
SA6612-5	NSA-030	NEED	12/07/07				
				ORG	1.0481	MG/L	(HIGH)

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TITLE: SAMPLE DISPOSAL

FIGURE 3

EXAMPLE OF HAZARDOUS WASTE STORAGE AREA DAILY CHECK

Daily Checklist for
HAZARDOUS WASTE STORAGE AREA

Week of: 1-28, 20 08

Item/Day:	Monday	Tuesday	Wednesday	Thursday	Friday
1. Are containers closed? (Except when waste is being added)	(Yes) / No	(Yes) / No	(Yes) / No	(Yes) / No	(Yes) / No
2. Are containers properly labeled with a hazardous waste label?	(Yes) / No	(Yes) / No	(Yes) / No	(Yes) / No	(Yes) / No
3. Do you have access to each container and can you read the label? (36" aisle?)	(Yes) / No	(Yes) / No	(Yes) / No	(Yes) / No	(Yes) / No
4. Is each container marked with the date storage began?	(Yes) / No	(Yes) / No	(Yes) / No	(Yes) / No	(Yes) / No
5. Are the dates on the containers less than 90 days old?	(Yes) / No	(Yes) / No	(Yes) / No	(Yes) / No	(Yes) / No
6. Is container free of dents, bulges, rust, spills or leaks?	(Yes) / No	(Yes) / No	(Yes) / No	(Yes) / No	(Yes) / No
7. Are all containers on a firm working surface?	(Yes) / No	(Yes) / No	(Yes) / No	(Yes) / No	(Yes) / No
8. Inspection by: Name (No Initials)	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
9. Time of Inspection	16:35	15:00	14:45	14:15	16:25
10. Verification of Inspection (Name/Date)	[illegible] 1-25-08	[illegible] 1-29-08	[illegible] 1-30-08	[illegible] 1-31-08 14:15	[illegible] 1-31-08 16:25

Deficiency noted:					
Corrective action:					
By (Name/Date):					

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TITLE: SAMPLE DISPOSAL

FIGURE 4

CHARACTERISTIC TOXIC HAZARDOUS WASTE AND TCLP CONCENTRATIONS

Chemical Name	CAS Number	Waste Code	TCLP conc. liquid	Equivalent conc. In Soil
Arsenic	7440-38-2	D004	5.0 mg/L	100 mg/kg
Barium	7440-39-3	D005	100 mg/L	2000 mg/kg
Cadmium	7440-43-9	D006	1.0 mg/L	20 mg/kg
Chromium	7440-47-3	D007	5.0 mg/L	100 mg/kg
Lead	7439-92-1	D008	5.0 mg/L	100 mg/kg
Mercury	7439-97-6	D009	0.2 mg/L	4 mg/kg
Selenium	7782-49-2	D010	1.0 mg/L	100 mg/kg
Silver	7440-22-4	D011	5.0 mg/L	20 mg/kg
Endrin	72-20-8	D012	0.02 mg/L	0.4 mg/kg
Lindane	58-89-9	D013	0.4 mg/L	8 mg/kg
Methoxychlor	72-43-5	D014	10 mg/L	200 mg/kg
Toxaphene	8001-35-2	D015	0.5 mg/L	10 mg/kg
2,4-D	94-75-7	D016	10 mg/L	200 mg/kg
2,4,5-TP (Silvex)	93-72-1	D017	1.0 mg/L	20 mg/kg
Benzene	71-43-2	D018	0.5 mg/L	10 mg/kg
Carbon Tetrachloride	56-23-5	D019	0.5 mg/L	10 mg/kg
Chlordane	57-74-9	D020	0.03 mg/L	0.6 mg/kg
Chlorobenzene	108-90-7	D021	100 mg/L	2000 mg/kg
Chloroform	67-66-3	D022	6.0 mg/L	120 mg/kg
o-Cresol	95-48-7	D023	200 mg/L	4000 mg/kg
m-Cresol	108-39-4	D024	200 mg/L	4000 mg/kg
p-Cresol	106-44-5	D025	200 mg/L	4000 mg/kg
Cresol	1319-77-3	D026	200 mg/L	4000 mg/kg
1,4-Dichlorobenzene	106-46-7	D027	7.5 mg/L	150 mg/kg
1,2-Dichloroethane	107-06-2	D028	0.5 mg/L	10 mg/kg
1,1-Dichloroethylene	75-35-4	D029	0.7 mg/L	14 mg/kg
2,4-Dinitrotoluene	121-14-2	D030	0.13 mg/L	2.6 mg/kg
Heptachlor	76-44-8	D031	0.008 mg/L	0.16 mg/kg
Hexachlorobenzene	118-74-1	D032	0.13 mg/L	2.6 mg/kg
Hexachlorobutadiene	87-68-3	D033	0.5 mg/L	10 mg/kg
Hexachloroethane	67-72-1	D034	3.0 mg/L	60 mg/kg
Methyl Ethyl Ketone	78-93-3	D035	200 mg/L	4000 mg/kg
Nitrobenzene	98-95-3	D036	2.0 mg/L	40 mg/kg
Pentachlorophenol	87-86-5	D037	100 mg/L	2000 mg/kg
Pyridine	110-86-1	D038	5.0 mg/L	100 mg/kg
Tetrachloroethylene	127-18-4	D039	0.7 mg/L	14 mg/kg
Trichloroethylene	79-01-6	D040	0.5 mg/L	10 mg/kg

FIGURE 4, cont'd

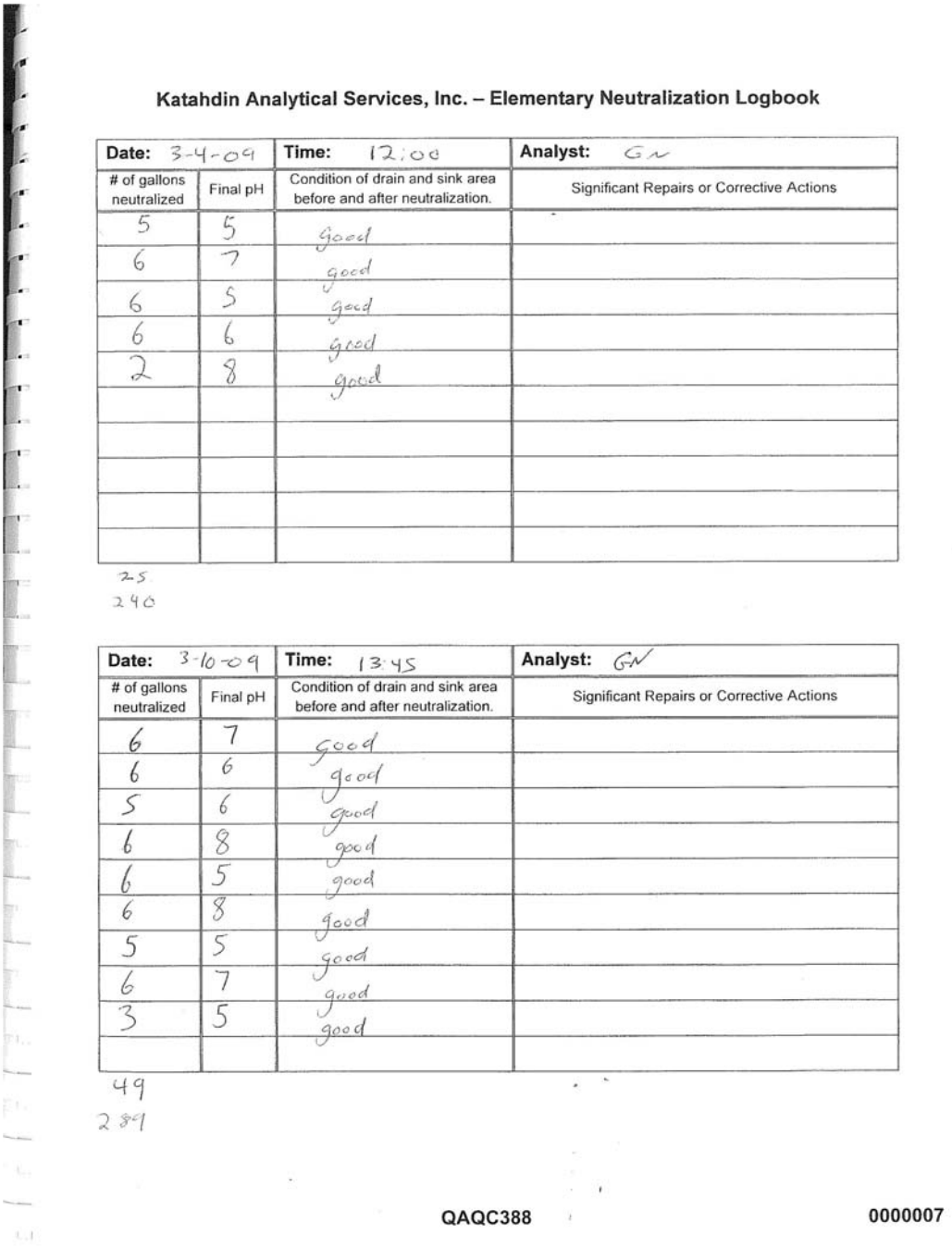
CHARACTERISTIC TOXIC HAZARDOUS WASTE AND TCLP CONCENTRATIONS

Chemical Name	CAS Number	Waste Code	TCLP conc. liquid	Equivalent conc. In Soil
2,4,5-Trichlorophenol	95-95-4	D041	400 mg/L	8000 mg/kg
2,4,6-Trichlorophenol	88-06-2	D042	2.0 mg/L	40 mg/kg
Vinyl Chloride	75-01-4	D043	0.2 mg/L	4.0 mg/kg

TITLE: SAMPLE DISPOSAL

FIGURE 5

EXAMPLE OF ELEMENTARY NEUTRALIZATION LOGBOOK



Katahdin Analytical Services, Inc. – Elementary Neutralization Logbook

Date: 3-4-09		**Time:** 12:00	**Analyst:** GN
# of gallons neutralized	Final pH	Condition of drain and sink area before and after neutralization.	Significant Repairs or Corrective Actions
5	5	good	
6	7	good	
6	5	good	
6	6	good	
2	8	good	

25
240

Date: 3-10-09		**Time:** 13:45	**Analyst:** GN
# of gallons neutralized	Final pH	Condition of drain and sink area before and after neutralization.	Significant Repairs or Corrective Actions
6	7	good	
6	6	good	
5	6	good	
6	8	good	
6	5	good	
6	8	good	
5	5	good	
6	7	good	
3	5	good	

49
289

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